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# BINDERS FOR COAL BRIQUETS

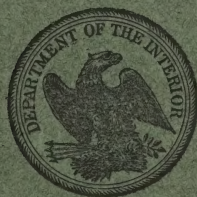
INVESTIGATIONS MADE AT THE  
FUEL-TESTING PLANT  
ST. LOUIS, MO.

BY

JAMES E. MILLS

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[Reprint of United States Geological Survey Bulletin 343]



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1911





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# BINDERS FOR COAL BRIQUETS:

## INVESTIGATIONS MADE AT THE FUEL-TESTING PLANT ST. LOUIS, MO.<sup>a</sup>

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By JAMES E. MILLS.

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### INTRODUCTION.

#### THE COMMERCIAL PROBLEM.

Coal, in the process of mining, transportation, and handling and on exposure to the weather, is subject to more or less disintegration. This disintegrated coal is usually called "slack" and amounts often to a considerable percentage of the lump coal produced in the mines. If this slack coal is wasted the loss so occasioned ranges from 5 to 50 per cent, or even more, of the total coal mined. It is therefore clear that the utilization of this waste slack coal becomes a serious economic consideration.

When the coal is suitable for the production of coke, the utilization of the slack presents no difficulty, as it is in demand for that purpose. If the coal does not produce good coke, but cakes rather readily, the slack can be used for boiler purposes, as it fuses together more or less quickly, and burns on the furnace grate without great loss. Coal that cakes less readily can be burned on grates of special construction. When so used it is more troublesome to handle, and the waste is greater than when lump coal is used. Consequently the price of much of the slack coal for fuel purposes ranges considerably lower than that of the lump coal from the same mine.

The full value of this slack coal as fuel can be realized by first forming the coal into a coherent mass or briquet, such briquets, when of good quality, being equal to or of greater value than the original

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<sup>a</sup> The writer undertook the work herein reported, in 1905, at the fuel-testing plant of the United States Geological Survey, under the direction of Dr. Joseph Hyde Pratt, of the University of North Carolina, to whom he is greatly indebted for advice and suggestions, given not alone at the beginning but throughout the progress of the work. Acknowledgment is also due for suggestions given by Mr. A. A. Steel, of the University of Arkansas, and for the assistance of many individuals and corporations who have answered inquiries and furnished samples as desired. In compiling this report and in laboratory work free use has been made of all available information thus acquired.

lump coal from which the slack was derived. The object of the investigations herein reported was to determine as far as possible to what extent the manufacture of briquets from slack coal may succeed commercially under the conditions existing in the United States.

The problem of briquetting is not always that of how to make the best possible briquet, for the slack at hand may be of inferior quality and the best possible binding material may be too expensive for the conditions prevailing in that particular locality. The problem is always to produce at a profit a briquet of satisfactory grade for the use intended. This problem will be made clearer by a brief summary of the available binders, followed by a preliminary discussion of the characteristics of a good briquet.

### THE KIND OF BINDER.

Definite answer to the question "What is the best binder to use in making briquets?" depends, as repeatedly emphasized in this paper, on the locality, on the character of the coal, and on the purpose for which the briquets are intended. For purposes of a brief comparison consideration is given to the binders available for a coal which is fairly easy to briquet and which cakes rather readily. A few coals will briquet with somewhat less and others require greater percentages of binder, but an endeavor has been made in the following summary to strike a reasonable average.

The experiments herein reported show that, in general, for plants situated where it can be obtained, the cheapest binder will prove to be the heavy residuum from petroleum, often known to the trade as asphalt. Four per cent of this binder being sufficient, its cost ranges from 45 to 60 cents per ton of briquets produced. This binder is particularly available in California, Texas, and adjacent territory.

Second in order of importance comes water-gas tar pitch. Five to six per cent usually proving sufficient, the cost of this binder ranges from 50 to 60 cents per ton of briquets produced. As water-gas pitch is also derived from petroleum, it will be available more particularly in oil-producing regions.

Third in order of importance is coal-tar pitch. Being derived from coal, this binder is very widely available. From 6.5 to 8 per cent will usually be required, and the cost ranges from 65 to 90 cents per ton of briquets produced.

Of local importance, where the price permits, are natural asphalts and tars derived from wood distillation. The price of each of these binders varies greatly with the locality, but there are doubtless places where they could compete with the binders above mentioned. Wax tailings could be used with an easily caking coal.

Pitch made from producer-gas tar is not yet on the market, but it will produce excellent briquets, with a lower percentage of binder



than other coal-tar pitches. It will doubtless be available in the future.

Briquets excellent in all respects except that they are not waterproof can be made by using 1 per cent of starch as a binder, the cost of which is 20 cents per ton of briquets produced. Extra care is necessary in drying and handling these briquets, and this adds to their cost.

The waste sulphite liquor from paper mills also produces excellent briquets except that they are not waterproof. At present it is a troublesome waste product dissolved in much water. Its utilization for this purpose will bear further investigation.

Of inorganic binders, magnesia might be utilized, as its probable cost would not exceed 22 to 30 cents per ton of briquets produced. Other inorganic binders, while available as regards price, would not make first-class briquets.

The briquetting of lignite coal offers a peculiarly difficult problem. If the lignite cakes in the fire, asphaltic residues from petroleum or water-gas tar pitch may be used as binder, larger percentages being required than for ordinary coals. The most promising binders for lignites that do not cake are starch, sulphite liquor, and magnesia. Lignites may be briquetted without binder if they are to be burned on grates specially constructed to overcome the tendency to fall to pieces in the fire.

Attention is called to the suggested method of deciding as to the value of coal-tar pitch for briquetting purposes. The method is likewise applicable to asphalts and petroleum residues generally: (1) The pitch or tar is distilled and all oils coming off below  $270^{\circ}\text{C}$ . are rejected as being of no value; (2) the flowing point of the portion to be used in briquetting is determined (this should generally not be less than  $70^{\circ}\text{C}$ .); (3) the pitch is extracted with carbon disulphide. The smaller the amount of residual carbon the more satisfactory is the pitch. The less readily the coal cakes the higher must be the flowing point of the pitch. If a pitch cracker is used, the pitch to work successfully on a hot summer's day must have a flowing point above  $120^{\circ}\text{C}$ . In the winter pitch with a flowing point of  $100^{\circ}\text{C}$ . may be used. All softer pitches and asphalts have to be melted and mixed in liquid form with the coal.

A pitch with a very high softening point, above  $150^{\circ}\text{C}$ ., should be either thinned or superheated in the mixer. The efficient use of a binder depends very largely on the proper regulation of the conditions in the mixer. The presence of low-volatile compounds in the pitch to be used as a binder increases the smoke in burning; and also increases the tendency of the briquet to soften and crack open in advance of combustion, owing to the volatilization and escape of these compounds.

The main problem in briquetting is to find a suitable binding material at sufficiently low cost. When the difference in price between the slack coal and the first-class lump coal is \$1, the cost of briquetting should not exceed this amount. Of this the binder must cost less than 60 cents per ton, as the cost of manufacture averages about 40 cents. To leave out of consideration the possible advantages in the use of briquetted coal over run-of-mine coal, due to the greater efficiency and smokelessness of briquets, it will probably not be necessary to pay any attention to binding materials costing \$1.25 or more per ton of briquets produced.

## CHARACTERISTICS OF GOOD BRIQUETS.

### COHERENCE.

The briquet should be sufficiently coherent. In France briquets are tested for coherence as follows:<sup>a</sup>

One hundred and ten pounds of briquets are divided into 100 pieces of 1.1 pounds each, which are placed in a cylinder 36.22 inches in diameter and 39.57 inches in length. This cylinder is divided into three compartments by diametrical partitions and revolves at a speed of 25 revolutions per minute. After being charged, it is revolved for two minutes, and the contents are thereupon sifted upon a screen perforated with holes 1.12 inches square. The proportion which does not pass through this screen indicates the degree of cohesive force, which, in the case of the French Admiralty tests, should reach 52 per cent, or if the fuel be intended for torpedo boat use, 58 per cent.

Briquets of any desired degree of coherence may be made by varying the amount of binding material used in the briquet and by varying the pressure. An increase of either the binder or the pressure, of course, represents an added cost in manufacture. Experiments made by M. Wéry, of Paris,<sup>b</sup> with a Biérix machine may be taken as illustrative:

*Effect on coherence of varying pressure and amount of binder.*

Pressure in kilograms per square centimeter.	Pressure in pounds per square inch.	Per cent of pitch used.	Per cent of cohesion obtained.
130	1,844	6	25
190	2,695	6	46
270	3,831	6	61
130	1,844	7	52
190	2,695	7	70
250	3,547	7	74

Ordinarily briquets may be considered sufficiently coherent when the loss occasioned by dust and breakage involved in their use does not exceed 5 per cent. Both manufacturers and consumers should recognize the desirability of adapting the briquet to the use intended.

<sup>a</sup> Briquets as fuel: Special Consular Report, vol. 26, p. 54.

<sup>b</sup> *Idem.*, p. 50.



### HARDNESS AND TOUGHNESS.

The briquet should be sufficiently hard; but if too hard it is likewise brittle, and therefore less coherent when subjected to rough handling. It is usually advantageous, therefore, to make the briquet of the minimum hardness that will suffice for the purpose in view. A briquet can be made harder by using a binder with a higher softening (melting) point. Consequently, if pitch is used, the most brittle pitch makes the hardest briquet. Moreover, a larger percentage of the more brittle pitch is usually required.

The requirement of the French Admiralty is that the briquet should not soften at 60° C. (140° F.). Ordinarily it is sufficient that the briquet shall not soften on the hottest day, and shall behave satisfactorily on burning.

### DENSITY.

It is sometimes specified that the briquet should have a density of not less than 1.19. Perhaps a better standard would require the briquet to about equal in density the lump coal from which the slack was derived, thus ranging from 1.1 to 1.4. The density is increased by pressure.

### SIZE AND SHAPE.

The convenience of a briquet for a given purpose, and hence the extent of its use, will depend largely on the size and shape. Attention is therefore called to the following points:

Heavy rectangular blocks allow a large output for the investment and are consequently cheaper to manufacture. They are convenient for storage. The French naval estimates show that 10 per cent more in weight of briquets can be stored in a given space than of lump coal, and the British Admiralty reports show a gain of as high as 20 per cent. Large rectangular briquets have the disadvantage of large smooth surfaces and are usually broken up when fed into furnaces, as this appears to promote combustion. To facilitate the breaking they are pressed with grooves or perforations. This gives better air circulation but decreases the output and the possibility of storage by just so much.

Prismatic shapes with rounded edges are most popular abroad. Either these or ovoid shapes of less than 2 pounds weight are preferred for domestic use. The rounded edges cause much less dust and breakage on handling and insure good air circulation and thorough combustion, but are wasteful in space and make the briquet somewhat harder to ignite.

The output of hollow, cylindrical, polygonal, and ball-shaped briquets abroad is small, the other shapes having proved more generally preferable.

## WEATHERING.

The briquet should stand long exposure to the weather with but little deterioration. A dense briquet will stand the weather better than a porous one. In the process of manufacture, briquets are liable to crack if they lack the proper proportion of binder, or if the binder and coal particles have been improperly mixed, or if the briquets are pressed too wet, or are insufficiently pressed. If the coal is finely ground, the briquet assumes a more dense and polished surface and is then more resistant to the weather. Cracks, however produced, allow the entrance of moisture and cause a rapid deterioration of the briquet on exposure to the weather. Lignite briquets, owing probably to the tendency of the lignite to absorb water and also to the more porous structure of the briquet, do not stand long exposure to the weather as successfully as other briquets.

The binder used must be insoluble in water. The great obstacle to the successful use of starch, molasses, and sulphite-liquor residues as binders is their solubility, the cost of rendering the briquet waterproof being usually prohibitive. It is deserving of serious consideration whether or not in certain dry portions of the West, where fuel is scarce, the waterproofing of the briquet could not be dispensed with altogether during the dry season, and to a considerable extent during the rainy season by keeping the briquets under cover.

With pitches, tars, etc., a slightly increased percentage of binder is necessary in briquets that are to stand long exposure to the weather. Further details are given under the discussion of the various binders.

## ABSORPTION.

The briquet should not absorb more than about 3 per cent of moisture. The amount of moisture absorbed is increased when either the slack itself or the briquet is porous, or when the binder used has a tendency to attract moisture.

## BURNING QUALITIES.

*Readiness of ignition.*—The ease with which a briquet will ignite depends largely on the slack used, but can be regulated to some extent. Large briquets ignite less readily than small ones. Sharp edges are an aid to ignition, though this advantage is not so great as to overcome the general preference for the prismatic and egg-shaped briquets. Briquets made from fine slack ignite less readily than those from coarser slack. A dense briquet is also more difficult to ignite. The use of an inorganic substance, such as clay or magnesia, as a binder, or as a constituent of the binder, tends to make the briquet ignite less readily. Increase of inorganic material—that is, ash—in the slack coal used produces the same result.



*Kind of flame.*—The briquet should burn with a clear, intense flame, and without odor or smoke. The burning of the briquet and the flame produced, as well as the smoke given off, will depend largely on the quality of the slack coal used and on the completeness of the combustion. The completeness of combustion can be regulated to some extent in the manufacture of briquets by making them of a shape to insure a good air circulation and by the choice of a suitable binder. So far as the choice of a binder for this purpose is concerned, the principle involved may be summed up in the statement that the smoke does not depend on the total amount of volatile matter in the briquet, but only on that part of the volatile matter which escapes before it is heated to the kindling temperature. In other words, the binder should not volatilize before the temperature is sufficiently high to insure complete combustion of the gases formed. In general terms, therefore, a binder adds smoke in proportion to the amount of low-boiling constituents (oils, etc.) that it contains.

Inorganic binders, of course, produce no smoke. Such organic binders as starch, molasses, or sulphite-liquor residues likewise do not volatilize until decomposed, and hence do not smoke, or smoke but little. Pitches, tars, and petroleum residues, when used as binders, volatilize, and will cause smoke and possibly odor if the gases formed are not completely burned. But it is quite possible to regulate the conditions, even when using these binders, in such a way that the briquets will produce less smoke than the lump coal from the screenings of which the briquet is made. This is due to the regular shape of the briquet, which allows a better-regulated air supply, enabling more complete combustion to take place. This reduction of the smoke nuisance is one of the advantages to be derived from the use of briquets.

*Retention of shape.*—The quality of retaining its shape in the fire is very important and depends on the properties of both the coal and the binder used in making the briquet. This point is discussed more fully in connection with the various coals and binders examined. The principle involved is very simple. The binder must hold the coal particles together until they are sufficiently softened to cohere. The temperature at which different coals soften or cake together varies greatly. Some bituminous coals cake readily at a low temperature, others less so. Semianthracite coals follow next in order, and then anthracite coals, some of the very hard anthracite coals with only a small amount of volatile matter showing little tendency to cake. Lignites as a class do not cake readily. Some, however, as those from Oklahoma or New Mexico, will cake sufficiently at a rather high temperature to hold themselves together. Others, as some California, Texas, or North Dakota lignites, show practically no tendency to soften or cake at any temperature. With such lig-

nites it is extremely difficult to make a briquet that will retain its shape in the fire. Briquets satisfactory for domestic use, when properly managed, can be made from such lignites. These briquets might be used in a variety of manufacturing operations if a grate suitably adapted to the fire box is provided. For use in a locomotive they would be less suitable.

With a readily caking coal, a binder that volatilizes (boils) at a comparatively low temperature may be used. With coals that cake at higher temperatures a less volatile binder must be used to obtain a satisfactory result in the fire. With a lignite that does not cake, the only binder that will enable the briquet to retain its shape until completely consumed is an inorganic binder which does not volatilize at all—unless, indeed, sufficient binder is added to practically coke the briquet. With such lignites, organic binders that do not volatilize, such as starch, molasses (in the form of waste residues from the sugar factories), sulphite-liquor residues from the paper mills, etc., give results that are fairly satisfactory, the briquet retaining its shape until the binder is itself decomposed. As the inorganic binders add ash and the other nonvolatile binders mentioned are not waterproof, it would seem generally better, where commercially possible, to mix a coal that will not cake of itself with a sufficient quantity of caking coal. Then when a suitable binder is used the briquet will retain its coherence in the fire by the softening of the caking coal used. The relation between the caking of a coal and its constitution is not well understood.

*Percentage of ash.*—The amount of ash left when the briquet is burned is the sum of that contained in the slack and in the binder used. Organic binders, as a rule, contain a smaller percentage of ash than the slack coal, and therefore slightly decrease the total percentage of ash in the briquet. When inorganic binders are used the ash thus added is a decided disadvantage.

In some foreign countries only 6 per cent of ash is permitted under many of the contracts for briquets. When the ash content of the slack exceeds 6 per cent it is therefore quite common abroad to wash the slack coal before briquetting. This saves freight on an incombustible material, saves binder, and gives in every way a better and more concentrated fuel. In this country, where good coal is so much cheaper than abroad, it will probably not usually prove feasible to wash the slack coal.

#### EVAPORATION RESULTS.

Theoretically the heating value of a briquet is the sum of the heating values of the coal and of the binder; and it can not possibly exceed this amount. Organic binders usually equal or exceed in heating value, weight for weight, the slack coal used. Usually,



therefore, they increase the total heat in a given weight of fuel, but owing to the small percentage of binder added, this increase is relatively slight. But the briquets have the advantage over the coal in that their burning is accompanied with less waste and they permit a better-regulated and more complete combustion to take place. In this way the heating value actually obtained from the fuel, weight for weight (and this, of course, is the important consideration), may be materially increased by the manufacture of the fuel into briquets. This increased heating value of the briquets over that of the slack used thus becomes a matter of practical importance.

The evaporation results should at least equal those of the best lump coal from the screenings and dust of which the briquet was made.

## CONDITIONS GOVERNING THE USE OF BINDERS.

### MAXIMUM COST ALLOWABLE FOR BINDER.

The output of a briquet plant depends to a very great extent on the size of the briquets manufactured. The cost of labor depends greatly on the size and arrangement of the plant and on the wages paid, which will vary considerably in different localities. The price of slack coal and of the different binders is even more dependent on the locality. An approximate idea of the total cost of manufacture, exclusive of the cost of the slack coal and the binder used, is here presented, in order to consider intelligently estimates which may be made of the maximum allowable cost of the binder, it being obviously useless to investigate a binder that could never be commercially used on account of its cost. E. Loze <sup>a</sup> estimates the cost for manufacture in France at 33 to 40 cents per ton. Schorr <sup>b</sup> states that the cost in France is 24 to 34 cents per ton; in Germany, 22 cents to 24 cents; and in England, 24 cents. Estimates of the cost in the eastern and western parts of the United States are as follows:

*Estimated cost per ton of manufacture of briquets in the United States (exclusive of binder and of coal briquetted).*

	Western States.	Eastern States.
Labor, inclusive of stacking.....	\$0.16	\$0.20
Oil and grease.....	.006	.01
Sundry stores.....	.01	.01
Steam (fuel).....	.04	.17
Depreciation.....	.05	.10
	.266	.49

Considering 30 to 50 cents per ton, therefore, as being approximately the cost of manufacture, it appears that when the difference in price

<sup>a</sup> Eng. and Min. Jour., vol. 76, 1903, pp. 277, 431.

<sup>b</sup> Trans. Am. Inst. Min. Eng., vol. 35, 1904, p. 100.

between the slack coal and the first-class lump coal is \$1, the binder must cost less than 50 to 70 cents per ton. Good briquets would probably find in many places a market at a price slightly advanced over that of the corresponding lump coal from the screenings of which the slack was derived. Yet it is evident that the main problem in briquetting is to find a suitable binding material at a cost sufficiently low. A binding material costing as much as \$1 per ton of briquets produced could be used profitably in but few places in the United States. Even allowing for future possible greater variation in price between the coal and the slack it is not necessary to pay attention to any binding material costing above \$1.25 per ton of briquets produced.

#### QUALITIES DESIRED IN BINDERS.

It is needless to say that a desirable binder should make a good briquet and should make it cheaply. The characteristics of a good briquet have already been pointed out. It will not, perhaps, be too great a repetition to summarize here, in the approximate order of their importance, the desirable qualities of a binder, as follows:

1. It must be sufficiently cheap to make the manufacture of briquets profitable.
2. It must bind strongly, producing a briquet sufficiently hard, but not too brittle.
3. It must hold the briquet together satisfactorily in the fire.
4. It must produce a briquet sufficiently waterproof to stand the conditions of use.
5. It should not cause smoke or foul smelling or corrosive gases, or foul the flues.
6. It should not increase the percentage of ash or clinker.
7. It should increase, or certainly not diminish, the heat units obtainable from a given weight of fuel.

#### EFFECT OF QUALITY OF BINDER ON THE BRIQUET.

##### SCOPE OF THE INVESTIGATIONS.

The behavior of a large number of different coals with a few binders and of a few coals with a large number of different binders has been very carefully studied. Tests were made with each coal and with each binder until the percentage of binder required to produce a satisfactory briquet with that coal was determined. The behavior of the briquets in the fire and, when necessary, in water was noted. The binders used were examined as to their chemical or physical properties and such modification of the binder was made as seemed likely to produce more efficient results.

The conclusions that follow are submitted as the net result of the studies thus outlined.



## PHYSICAL RELATION OF COAL AND BINDER.

The relation between the coal and the binder is purely physical. Chemical action, if coming into play at all, is so slight in amount as to be wholly negligible. Moreover, the properties of the binder are not greatly changed by the mutual solubility, or surface action, of coal and binder at the surface of the coal.

The above statements are shown to be true by the fact that if the coals are arranged in a series according to the percentage of one binder required, they will retain that same order when other binders are used, even when these binders are of the most diverse nature. The experiments of Constam and Rougeot<sup>a</sup> show that the soluble portion of the binders (various pitches) could be extracted from the briquets practically quantitatively with carbon disulphide, and that this reagent extracted at the most only 0.7 per cent from the coal.

The properties of the briquet are the properties of the coal plus the properties of the binder, and the combination of the two in briquetting does not materially change the properties of either. Not only is this observation true of briquets at ordinary temperatures, but it is also confirmed by their behavior in the fire. The decomposition of the binder caused by the heat may alter its character to some extent, but never, so far as the writer has observed, sufficiently to mask its original character. The action of the briquet in air and in water also confirms the truth of the above observation.

## QUALITIES OF BINDER IMPARTED TO BRIQUET.

If the binder is brittle the briquet will be relatively brittle at the same temperature. Thus rosin, hard pitches, asphalts, cements, etc., make briquets that are hard, but they break easily from a sharp blow or fall. Liquids such as coal tar, creosote, asphalt tar, etc., make briquets that do not break easily from a fall, but they yield so readily to pressure as to be useless. Comparable percentages of binder being used, the toughest briquet—that is to say, the briquet that will stand the most rough usage—is made with a binder that at ordinary temperature twists easily and pulls into threads, that will cut with a knife rather than break, and that flows very slowly, taking some time to assume the shape of the container. Such a binder is sufficiently elastic not to be brittle and is sufficiently stiff not to yield to climatic changes of temperature. Binders that have been examined fulfilling this condition are pine-wood tar (12),<sup>b</sup> water-gas tar pitch (39), wax tailings (40), and residuums from petroleum, often designated as asphalts (37 A, 37 B, and 37 C). Satisfactory briquets are made with 3 to 5 per cent of the above binders. If the coal does

<sup>a</sup> Zeitschr. f. angew. Chemie, vol. 17, No. 26, p. 1.

<sup>b</sup> Numbers refer to list on p. 22.

not cake readily a binder with a higher melting point would be required to make the briquet retain its shape in the fire.

#### BEHAVIOR WHEN HEATED.

The binder will soften when in the briquet as soon as it is heated to the temperature at which it softens when outside of the briquet. Such softening will not be so apparent, however, for the binder exists in the briquet as a very thin coating over the grains, and if it melts to a thick, sticky liquid, rather than to a limpid one, its cohesive power in the state of a liquid is still very great. But it must be borne in mind that all briquets have a temperature of maximum weakness in the fire. This temperature lies in the interval between the melting or destruction of the binder and the softening of the coal as it commences to cake. If the coal softens at a high temperature the binder must melt at a relatively high temperature to give satisfactory results in the fire. If the coal does not cake at all, then the binder must not melt at all, or be destroyed by the heat, if a perfectly coherent briquet at all temperatures is desired. Only inorganic binders could fulfill this condition, and their use is objectionable. Organic binders that do not melt, such as starch, etc., give the best results in the fire with a noncaking coal, but are not waterproof.

In a furnace the briquet does not become thoroughly heated throughout at the same time, and as the binder near the surface of the briquet melts and passes out as a gas, the binder in the next interior layer of the briquet to some extent takes its place, and so on. In this way the briquet is held together until the coal at its surface softens and cakes. When this happens the briquet commences to regain its strength and with many coals soon becomes stronger than when placed in the fire.

The binder will volatilize out of the briquet and appear as a gas as soon as it reaches the temperature at which it boils when outside of the briquet and in the pure condition. If this happens much below the kindling temperature of the gas some smoke and odor will be caused, and the smoke and odor may to a large extent be taken as proportional to the low-boiling oils in the binder—at least so far as the smoke is caused by the binder and not by the coal.

#### SOLUBILITY.

If the binder used is to any extent soluble in water the briquet will not withstand exposure to wet weather. The binder will go into solution as surely, though more slowly, in the briquet, as when it exists in the pure condition outside of the briquet, unless the briquet is in some way rendered waterproof.



# QUANTITY OF BINDER NECESSARY.

## SURFACE TO BE COATED.

The fact that the binder exists unchanged in the briquet, its office being solely to coat the grains, fill up void spaces between the grains, and by its adhesive and cohesive properties hold the briquet together, points to the following conclusions.

The amount of binder required will depend on the amount of surface to be coated, and the amount of surface will depend on the size of the grains, on their density (that is, the density of the dry coal), and on the capillary pores in the coal. The theoretical relation between the amount of surface to be coated, the size of the grains, and the density of the coal can be easily computed.

Let  $w$  = weight of coal taken. Suppose the grains of coal to be spheres, and let  $r$  = radius of the sphere. Let  $d$  = density of the coal. Then the volume of the sphere is  $\frac{4}{3} \pi r^3$ . The weight of the sphere is  $\frac{4}{3} \pi r^3 d$ . The number of grains of coal in the weight of coal taken is  $\frac{w}{\frac{4}{3} \pi r^3 d}$ . The surface of each grain is  $4 \pi r^2$ , and the total surface to be coated is  $\frac{3w}{rd}$ .

That is, the amount of surface to be coated varies inversely with the density of the coal and inversely with the diameter of the grains. The same law can be shown to apply whatever the shape of the grains.

The practical bearing of this relation is important. Thus, suppose a coal of density 1.4 requires 6 per cent of pitch to make a satisfactory briquet. Then a coal of density 1.1, other things being the same, would require 7.63 per cent of pitch, or 1.63 per cent more pitch than is required by the denser coal. This is one reason why lignite coal with a low specific gravity requires more binder than the average coal.

The variation in the size of the grains of coal has an even greater influence on the amount of binder required. The table below shows the relative amount of surface to be coated in coal slack of varying degrees of fineness:

*Relation between size of grains and amount of surface.*

Number of meshes to inch.	Diameter of wire (inch).	Size of mesh (millimeters).	Relative amount of surface.	Number of meshes to inch.	Diameter of wire (inch).	Size of mesh (millimeters).	Relative amount of surface.
1.....	0.131	25.400	1	80.....	0.00575	0.230	110
2.....	.103	12.700	2	100.....	.00450	.170	150
4.....	.079	6.350	4	200.....	.00235	.085	300
10.....	.027	2.000	12.7	.....	.....	.005	5,080
20.....	.01650	1.000	25.4	.....	.....	.0025	10,160
30.....	.01375	.670	37.9	.....	.....	.00075	33,900
40.....	.01025	.500	50.8	.....	.....	.00025	101,600
50.....	.00900	.310	81.9	.....	.....	.....	.....

It will thus be seen that coal slack which will just pass a 20-mesh sieve has 6.35 times as much surface to be coated as the same weight of slack crushed so as to pass a screen of  $\frac{1}{4}$ -inch mesh, and that coal passing a 200-mesh sieve has 75 times the surface of coal just passing the  $\frac{1}{4}$ -inch mesh. The very finest dust, having a diameter of 0.00025 millimeter, has 25,400 times the surface of coal just passing the  $\frac{1}{4}$ -inch mesh.

This consideration is not purely theoretical. The remark of Wagner,<sup>a</sup> that it took 20 per cent of pitch to briquet certain fine coal dust, is illustrative of its practical bearing. The degree of fineness of the slack coal used is one of the main factors in determining the percentage of binder necessary to produce a satisfactory briquet.

To illustrate this point, mention is here made of a fact shown later, that all coal-tar pitches contain a certain amount of carbon (soot), which, being in a very finely divided condition, is not only inert so far as binding the coal together is concerned, but itself requires a binder. Owing to the dustlike condition of this carbon its effect on the binding power of the pitch for the coal is most marked. Thus, although a coal-tar pitch (28 G) that contained 14 per cent of this inert, finely divided carbon made a satisfactory briquet with Illinois No. 4 coal when 6 per cent of the pitch was used, yet another coal-tar pitch (28 I) containing 37 per cent of the inert carbon failed to make a satisfactory briquet with the same coal when 14 per cent of the pitch was used. On the market the pitches sell at approximately the same price. The serious mistake made in crushing coal slack too fine is apparent.

Fine crushing of the coal slack gives the briquet a smoother surface that is more resistant to the weather; but this increase in the quality of the briquet is usually obtained at too great a cost, owing to the additional binder required, as explained above. Fine crushing also makes the briquet somewhat harder to ignite.

Capillary pores increase the amount of surface to be coated and the amount of void space to be filled, and this is probably another reason why lignites require more binder than hard coals.

It is interesting, in this connection, to note that with all binders the coherence in the briquets at first increases but slowly with increase in the proportion of binder. Then suddenly the coherence increases very rapidly and the briquets become strong. Then when an excess of binder is added the increase in strength is again only slight. The curve takes the form indicated in the accompanying diagram (fig. 1). The explanation, of course, lies in the fact that at first there is not enough binder to coat all the grains of coal and there can be little coherence. When sufficient binder has been added to coat the grains, the strength increases rapidly. After the grains have been well

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<sup>a</sup> Cassier's Magazine, vol. 11, 1896, p. 23.



coated there is little further gain in strength with the use of additional binder.

#### PERCENTAGE OF VOIDS.

The amount of binder will depend on the amount of void space to be filled. There should always be enough of the finer coal and coal dust present to fill the spaces between the larger grains, or binder will be required to fill these spaces. Thus Wagner also found that a very large amount of binder was required to bind coal slack of a uniform size, five-sixteenths to three-eighths inch in diameter. Clifford Richardson, in a recent book on "Modern asphalt pavements," gives a calculation by Dr. G. F. Becker, of the United States Geological

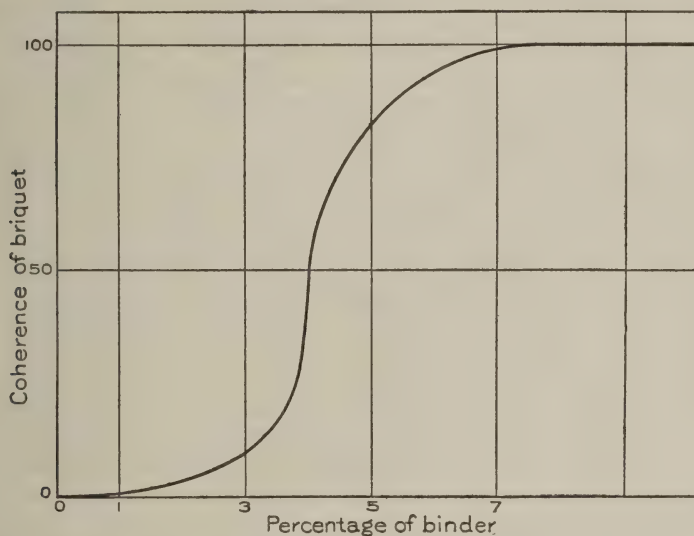


FIG. 1.—Curve showing relation between percentage of binder (water-gas tar pitch) and coherence of briquet. Other binders show similar curves, but with different percentages.

Survey, as to the amount of void space. This calculation is in outline as follows:

Consider four spheres in a plane so arranged that the lines joining their centers form a square, and four other spheres above them. A cube is formed by the lines joining the centers of the eight spheres. If  $r$  is the radius of a sphere, then the volume of the cube is  $8 r^3$  and the void space is  $8 r^3 - \frac{4}{3} \pi r^3$ , and the percentage of void space is  $\frac{8 r^3 - \frac{4}{3} \pi r^3}{8 r^3} = 1 - \frac{\pi}{6} = 0.4764$ . If the spheres are placed obliquely, then the area of the parallelogram joining their centers is  $2 r^2 \sqrt{3}$ , and multiplying this by the height of a tetrahedron formed by the centers of four spheres when three are placed in contact in one plane

and the fourth is placed on them, we have for the volume of the prism  $4 \sqrt{2} r^3$ . Then for the percentage of voids we will have

$$\frac{4 \sqrt{2} r^3 - \frac{4}{3} \pi r^3}{4 \sqrt{2} r^3} = 1 - \frac{\pi}{3 \sqrt{2}} = 0.2595.$$

From these results it will be seen that the amount of void space between grains of uniform size is independent of the size of the grains. In practice, however, even shot will not pack quite so closely as the theory indicates, as is shown by the experiments of Richardson, who found that with shot the percentage of void space was about 32.<sup>a</sup>

With grains of sand of uniform size but of irregular shape Richardson found the void space to average 43.6 per cent. It may be said, therefore, that in briquetting coal, 56.4 per cent of the total weight of the slack should be in grains about one-fourth inch in diameter.

It is interesting to obtain some idea of the desirable fineness of the remaining coal particles. Without giving the calculation in detail we may say that theoretically the spheres fitting in the spaces between the larger spheres, and the yet smaller spheres fitting into the void places then left can be calculated. The calculation shows that if  $r$  represents the radius of the large sphere there would be for every large sphere one smaller sphere having a radius of .4142  $r$ , two spheres having a radius .2247  $r$ , five spheres having a radius .1763  $r$ , and eight spheres having a radius .1543  $r$ . The volume occupied by these smaller spheres will be 11.14 per cent of the total volume, and since the large spheres occupy 74.05 per cent of the total volume, we would have about 15 per cent of void space to be filled in by yet smaller spheres. With irregular grains the results would not follow the theoretical percentages; but in a general way it is apparent that although it is advantageous to have a large percentage of the grains coarse (say 60 per cent of  $\frac{1}{4}$ -inch diameter), yet a considerable amount (say 40 per cent passing a 20-mesh sieve) of the finer slack and dust must be present to fill the voids.

The coal used in briquetting being already for the most part fine slack, the best practical results will be obtained by not breaking any of the lumps that are larger than one-fourth inch in diameter more than is necessary to bring them to that diameter and by not crushing the finer coal at all.

#### THICKNESS OF COATING.

The amount of binder necessary will depend on the thickness of the coat of binder over the surface of the grains of coal. The thickness of the coat of binder required will vary both with the coal and the

<sup>a</sup> This is partly accounted for by the fact that the discussion of Doctor Becker does not consider the contact of the spheres with the walls of the container.—J. E. M.



binder, but principally with the binder. In general, it may be said that the binder should be dissolved or heated until it is in the condition of a thin liquid capable of wetting the grains, somewhat as water would. With the harder pitches or asphalts, and similar binders, superheated steam for the mixers is a matter of necessity for economical working, for otherwise the binder does not become sufficiently liquid to spread in a thin coat and is therefore wasted.

#### OTHER CONSIDERATIONS.

The amount of binder required will depend to a slight extent on that portion of the coal which, being soluble in carbon disulphide, may be regarded as "bitumen" and as having some binding power. Constam and Rougeot<sup>a</sup> never found the amount of carbon disulphide extract to exceed 0.7 per cent, and probably with most coals the amount is negligible.

If the coals are arranged in a series according to the percentage of one binder required they will retain that same order in the series when other binders are used. Furthermore, if the equivalent percentages of different binders are determined for one coal then these equivalent percentages can be used for all coals, slight modifications only being sometimes necessary. An advantageous arrangement would be to place coals as ordinates and binders as abscissas in a table, and then the percentages of any binder required with any coal could be read directly.

### LABORATORY INVESTIGATIONS OF VARIOUS BINDERS.

#### METHODS AND SCOPE OF THE EXAMINATION.

##### DETERMINATION OF PERCENTAGE OF BINDER.

In order to determine in the laboratory the percentage of pitch necessary to briquet a given coal, 20 grams of coal, unless otherwise stated in the detailed report, was weighed out, mixed with the chosen percentage of binder, and placed in a Battersea crucible. A small amount of water was then added and the mixture heated, with sufficient stirring to mix the binder and coal thoroughly, until steam came off freely and only a small amount of water was left in the coal. The mixture while still hot was pressed in a small laboratory hand press, on which a pressure of 3,500 to 4,000 pounds per square inch was usually obtained. Each briquet made weighed about 5 grams, and thus four briquets were obtained as representing the test. The percentage of binder was varied in subsequent tests until the correct percentage to produce a satisfactory briquet was determined.

The percentage of binder was always calculated on the weight of the coal, consequently the percentage calculated on the weight of the

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<sup>a</sup> Zeitschr. f. angew. Chemie, vol. 17, No. 26, p. 1.

briquet produced would be somewhat less. This is a matter of no consequence, however, as the method of grading the briquet was purely relative.

#### DETERMINATION OF COHERENCE.

The examination of the small briquets produced was somewhat crude—their coherence being determined by the way in which they crushed or broke. The briquets were graded by numbers as follows:

- |                                    |  |
|------------------------------------|--|
| 1. Very slight coherence.          | 4½. Excellent briquet; would stand rough handling. |
| 2. Slight coherence.               |  |
| 3. Coherent, but not satisfactory. |  |
| 4. Satisfactory.                   |  |
|                                    | 5. A briquet stronger than necessary.              |

It was found somewhat difficult to compare extremely hard and brittle briquets with others not brittle but too soft. In all tests the intention was to produce a relative grading in which 4 would represent a satisfactory briquet for ordinary use. In actual work the coherence of the briquet could be varied to suit the demand of the customer, but in no case probably would such variation exceed the range represented by the numbers 3½ to 4½.

#### LIST OF MATERIALS STUDIED.

The materials used to bind the particles of coal together may be either organic or inorganic, and a very large number of substances have at various times been suggested and used for this purpose.

A list of the binders which have been examined is given below. An effort has been made to include in this list all binders which it was thought might be used commercially in the United States, as well as certain other substances which seemed fitted to throw light on the laws governing the action of the binder. Attempt was made to study such modifications and combinations of the different binders as it seemed might produce more efficient commercial results. For these latter modifications and combinations reference must be had to the detailed report.

#### INORGANIC BINDERS.

(1) Clay, (2) lime, (3) magnesia, (4) magnesia cement (magnesium oxide and magnesium chloride), (5) plaster of Paris, (6) Portland cement, (7) natural cement, (8) slag cement, (9) water glass.

#### ORGANIC BINDERS.

*Wood products.*—(10) Rosin, (11) pitch (rosin and tar), (12) pine-wood tar, (13) hard-wood tar, (14) Douglas fir tar, (15) wood pulp, (16) sulphite liquor (from paper mills).

*Sugar-factory residues.*—(17) Beet pulp, (18) lime cake, (19) beet-sugar molasses, (20) cane-sugar molasses.

*Starch.*—(21) Corn starch, (22) potato starch.

*Slaughter-house refuse.*



*Tars and pitches from coal.*—(23) Blast-furnace tar, (24) producer-gas tar, (25) illuminating-gas tar, (26) by-product coke-oven tar, (27) coal-tar creosote, (28) various grades of pitches from various tars.

*Natural asphalts.*—(30) Impsonite, (31) gilsonite, (32) maltha, (33) refined Trinidad, (34) refined Bermudez, (35) hard and refined asphalts (from impregnated sandstones, etc.).

*Petroleum products.*—(36) Crude oil, (37) residuum (asphalts, etc.), (38) water-gas tar, (39) water-gas tar pitch, (40) wax tailings, (41) acid sludge, (42) asphalt tar, (43) Pintsch gas tar, (44) Pittsburg flux.

## INORGANIC BINDERS.

### GENERAL STATEMENT.

The great disadvantage of inorganic binders is that they all add ash to the fuel. This means freight on just so much noncombustible material, less heat return for a given weight of fuel consumed, and an added amount of ash on the grate. All briquets made with inorganic binders are weak when first pressed and strengthen only gradually. Inorganic binders possess the advantage that they are not volatile, and hence the briquets, even when made from a noncaking coal or lignite, will stand up well in the fire without disintegration. They also have a tendency to lessen the smoke produced. This is due to the fact that the binder enables a somewhat slower and more complete combustion to take place and does not itself contribute any smoke to the fuel.

Another slight advantage sometimes claimed for certain of the inorganic binders, such as lime, water glass, and magnesia, results from the tendency of the calcium, sodium, and magnesium to combine with the sulphur, thus diminishing the escape of the sometimes objectionable oxidation products of that substance. This action would be the same if the calcium, etc., existed in the binder in chemical combination, as it occurs in calcium resinate. (See "Rosin," p. 30.) For the purpose of testing the above-mentioned claim, a briquet was made with Indiana No. 8 coal and 4 per cent of magnesium oxide. The briquet was dried and then burned. The sulphur in the ash (determined by the kindness of Mr. Somermeier) was found to amount to 0.44 per cent. As the sulphur in the coal was 3.72 per cent, it is evident that only a small fraction of the sulphur is retained by the magnesium oxide used as a binder. The same would probably also hold true for calcium and sodium compounds. It is thought, therefore, that the advantage thus gained is not great enough to merit consideration in practice.

Evidently the disadvantage resulting from the addition of any large percentage of an inorganic binder is too great to justify its use except as a matter of great saving in cost, or as a matter of necessity, in order to hold together in the fire some entirely noncaking coal and produce a low grade of fuel therefrom.

The essential results of the tests made with the different coals and binders are assembled in the table at the end of this report, wherein is shown the percentage of binder necessary to produce a satisfactory briquet with the coal considered.

The work of the laboratory can be regarded as sufficient so far as the negative results are concerned, but in all cases where the laboratory work seemed to promise commercial results the experiments should be repeated on a larger scale.

The inorganic substances which were tested are the only inorganic materials whose use as a binder on a commercial scale seemed even so remotely possible as to warrant testing in the laboratory. A list of other inorganic substances which have been suggested as binders, or as possible constituents of binders, would include chalk, alum, ammonium chloride (sal ammoniac), copper sulphate, sodium hydroxide, sulphur, potassium nitrate, calcium chloride, etc. That all these substances are totally unfit for such purpose appears at once from a knowledge of their properties, and they were not considered further.

#### DETAILED DESCRIPTION.

1. *Clay*.—The tests shown in the table (pp. 51–52) were made with a good sample of potter's clay obtained through Dr. J. H. Pratt. Clay is cheaper than coal and its cost, considered as a binder, is therefore a minus quantity.

The briquets when first taken from the press were extremely weak, many of them breaking while being taken out. The full pressure could not be given, for the coal would crush through the narrow, practically closed space between the molds and the bed plate. After drying, the briquets were hard and rather brittle. In water they fell to pieces completely and quickly. In the fire they hardened and stood up well, except those made of the noncaking lignite, California No. 1, which nevertheless stood up far better than with most binders and in comparison with the usual behavior of this lignite could be called very satisfactory.

Clay was used as a binder at one of the first plants established in this country, the Loiseau plant at Port Richmond, Pa. Trouble was experienced with the press used, the briquets when first made showing weakness. This was finally overcome, but the binder was abandoned owing to the expense of drying and waterproofing the product. Briquets made at this plant with clay were said to be very satisfactory in the fire.

Any press using clay for a binder would probably have to be specially adjusted. Owing to the large addition of ash, and to the expense of drying and waterproofing the briquet, it is improbable that clay will ever prove advantageous as a binder. If used alone it



can only be for the manufacture of a poor grade of fuel, incapable of standing any exposure to rain.

Clay in connection with other binders may be regarded as an adulteration of very doubtful value to the consumer.

2. *Lime*.—Lime, or rather, milk of lime,  $\text{Ca}(\text{OH})_2$ , has often been suggested as a binder, and is said to have been used. The tests shown in the table were made with calcium oxide known to be chemically pure. In these tests the lime was mixed dry with the coal, and then water was added. In some of the tests an excess of water was added and later boiled off; in others an excess of water was added and then squeezed out in the press; and in yet others only sufficient water was added to thoroughly moisten the mass. After drying, all the briquets were very weak, those in which the largest percentage of calcium oxide was used being the worst. They finally disintegrated, merely from exposure to the air.

From these tests it is difficult to understand how it is possible to use lime alone to make a briquet. For further experiments with lime see "Rosin," (p. 30).

3. *Magnesia*.—The sample of magnesia tested was a light, calcined magnesium oxide. In the tests shown in the table (pp. 51–52) the magnesium oxide was mixed with the coal and then a sufficient amount of water was added. In some tests the briquet was pressed cold and in others more or less of the water was evaporated. The results show that 3 to 5 per cent of this binder would make a satisfactory briquet, except with certain lignites. The briquets are very hard and would stand heavy pressure, but are brittle if less than 4 per cent of binder is used. In water the briquets go to pieces, though far less rapidly than those made with clay. In the fire they behaved very well, some being satisfactory even when only 2 per cent of binder was used.

In the United States magnesite, from which magnesia is obtained, is found only in California, where the production of magnesium oxide in recent years has been as follows:

*Quantity and value of magnesia produced in the United States, 1901–1906.*

Year.	Quantity.	Value per ton. <sup>a</sup>	Year.	Quantity.	Value per ton. <sup>a</sup>
	<i>Short tons.</i>			<i>Short tons.</i>	
1901.....	1,666	\$7.56	1904.....	1,357	\$8.22
1902.....	1,349	7.56	1905.....	1,873	9.76
1903.....	1,750	7.27	1906.....	3,714	7.56

<sup>a</sup> Based on value of raw magnesite, with 10 per cent added to cover cost of manufacture of magnesium oxide therefrom, being a suggestive approximation only.

The production could be greatly increased, several million tons of the magnesite being now in sight. The mineral is calcined for the

production of carbon dioxide, leaving the magnesia, which is used principally for covering steam and heating pipes, by paper mills, and in the manufacture of bricks for lining open-hearth furnaces and converters.

At the price prevailing in 1903, the cost of 3 per cent of this binder would be about 22 cents per ton of briquets produced. Three or four per cent of ash added to the fuel would not be greatly injurious, and the binder would possess an advantage over organic binders in holding the briquet together in the fire and in reducing the smoke.

The claim that the magnesia in the briquet reduces the amount of sulphur that escapes from the coal, as already pointed out (p. 23), seems to be of no practical importance.

It seemed possible that coke breeze might be briquetted with this binder, the briquets to be used in the place of coke in the furnace. Laboratory experiments on this point, however, gave unsatisfactory results, as follows:

*Results of briquetting coke breeze with magnesia.*

Percent- age of binder.	Grade of coher- ence. <sup>a</sup>
3	3
4	3
6	4
8	4½

<sup>a</sup> See p. 22.

In water the briquet with 6 per cent of magnesia behaved fairly well and that with 8 per cent splendidly, but in the fire the briquet with 4 per cent was unsatisfactory, that with 6 per cent was only fair, and that with 8 per cent was very hard to ignite.

For results of experiments with mixtures of magnesia and organic binders see p. 49.

4. *Magnesia cement*.—In 1880 Dr. A. Gurlt recommended a binding material consisting of 30 parts of 45 per cent magnesium chloride, 30 parts of 93 per cent magnesium oxide, and 60 parts of water. He used 5 per cent of this material and says that it produced a stronger briquet than any other and that it adds only 2.5 per cent of ash. The statement as to the amount of ash (magnesium oxide) added is correct. The formula on examination, leaving out the water, is found to reduce to  $5\text{MgO}.\text{MgCl}_2$ . The evidence on which this formula was taken as the most advantageous for the cement is not stated. The results reported in the following table are based on the proportions shown for the formulas therein given:



*Results of briquetting Illinois No. 11 B coal<sup>a</sup> with varying formulas of binder.*

Formula.	Calculated for 2 per cent of magnesium oxide in the ash.			Calculated for 3 per cent of magnesium oxide in the ash.			Remarks.
	Amount of binder per gram of coal.		Grade of coher- ence. <sup>b</sup>	Amount of binder per gram of coal.		Grade of coher- ence. <sup>a</sup>	
	MgO.	MgCl <sub>2</sub> 6H <sub>2</sub> O.		MgO.	MgCl <sub>2</sub> 6H <sub>2</sub> O.		
	<i>Gram.</i>	<i>Gram.</i>		<i>Gram.</i>	<i>Gram.</i>		
MgO.MgCl <sub>2</sub> .....	0.0100	0.0500	2½	0.0150	0.0750	3	} Stronger than preced- ing.
2MgO.MgCl <sub>2</sub> .....	.0133	.0334	2½	.0200	.0500	3	
3MgO.MgCl <sub>2</sub> .....	.0150	.0250	2½	.0225	.0375	3	
4MgO.MgCl <sub>2</sub> .....	.0160	.0200	2½	.0240	.0300	3	
5MgO.MgCl <sub>2</sub> .....	.0167	.0167	3	.0250	.0250	3½	} Apparently of about equal strength.
6MgO.MgCl <sub>2</sub> .....	.0171	.0145	3	.0256	.0216	3½	
7MgO.MgCl <sub>2</sub> .....	.0175	.0127	3	.0262	.0190	3½	
MgO.....	.0200	.0000	3	.0300	.0000	3½	

<sup>a</sup> Bituminous coal (one-half run of mine, one-half lump) from shaft near Cartersville, Williamson County, Ill. For description, analysis, and tests see Bull. U. S. Geol. Survey No. 290, 1906.

<sup>b</sup> See explanation under "Determination of coherence" (p. 22).

In these tests the magnesium oxide was mixed dry with the coal, and then the magnesium chloride (dissolved in water) was added. As already stated, the method of testing the small briquets made does not allow of minute differences being noted, but the results showed clearly an increase of strength until the proportion given by Doctor Gurlt and represented by the formula 5MgO.MgCl<sub>2</sub> was reached. On still further decreasing the proportion of the magnesium chloride the briquets apparently did not grow either weaker or stronger. Magnesium oxide is cheaper than the chloride, and in view of the results obtained there is considerable doubt as to the advantage of adding the chloride. The addition of the chloride is said to make a more quickly setting cement, and one that is more insoluble, owing to the formation of an oxychloride of magnesium, but the statement is not verified. The magnesium chloride would also have the disadvantage of losing its chlorine in the fire, and this might come off either free or combined with hydrogen as hydrochloric (muriatic) acid. In either case the resulting gas is exceedingly corrosive and would greatly injure the boiler flues. Possibly all of the chlorine would be retained by the coal ashes, but it is a matter of grave doubt.

In the fire briquets made with 3 per cent of magnesia cement of the formula 5MgO.MgCl<sub>2</sub>—that is, 3 per cent after calculating the formula to MgO—stood up well. In water they disintegrated after some time. It was not evident that the briquets with magnesia cement of this formula behaved any better in water than briquets made with the same ash percentage of magnesium oxide alone, if indeed they behaved so well.

Magnesium chloride is ordinarily sold in the market in the crystallized form MgCl<sub>2</sub>.6H<sub>2</sub>O. This grade is quoted at \$20 per ton in large lots in New York. It is not produced to any considerable extent in this country, but should the demand arise could probably be made from the California magnesite without increasing the cost.

All the briquets made with the magnesia cement were very hard but very brittle. They would stand great pressure, but apparently would not stand rough handling, when only 5 per cent of the cement is used, as recommended by Doctor Gurlt.

5. *Plaster of Paris*.—Gypsum, the mineral from which plaster of Paris is produced, is widely distributed in the United States. In 1903 the production was 264,196 tons, valued at \$4.08 per ton.

The tests shown in the table (p. 51) were made with plaster of Paris which was first mixed with the coal. Sufficient water was added to thoroughly moisten the mass, and then pressure was applied, the excess of water, if any, running out in the press. The briquets were very hard, but also brittle, and would not stand rough handling unless at least 12 per cent of binder was used. Even these were not first-class briquets. In the fire the briquet with 12 per cent of binder held together perfectly, and would have held together with a smaller percentage. In water the briquet went to pieces more rapidly than was expected.

Although even 12 per cent of plaster of Paris in a briquet would not be prohibitive as regards cost (50 cents per ton of briquets produced), it would be as regards the addition of ash, and would moreover cause a much slower combustion of the briquet. A briquet with 6 per cent shows considerable coherence and might be satisfactory for some purposes. For results of experiments with mixtures of plaster of Paris and organic binders see page 49.

6. *Portland cement*.—In 1903, 22,342,973 barrels of Portland cement, weighing 400 pounds gross each, were produced in the United States. The average value per barrel was \$1.24, and allowing 20 pounds tare for the barrel, the value per ton was \$6.52.

The sample of Portland cement tested was obtained from Mr. Richard L. Humphrey and was a mixture of seven well-known brands, constituting what has been termed typical cement. In the first tests made the cement was mixed with the coal, then an excess of water was added and largely boiled off, after which the coal was pressed. The results not being satisfactory, in subsequent tests less water was added and the mixture was not heated, but the results were only a little better. In the fire briquets with 12 per cent of binder held together well, and a smaller percentage would have been sufficient. In water the briquets went to pieces somewhat more rapidly than those made with plaster of Paris.

This binder is more expensive and certainly no better than plaster of Paris. For results of experiments with mixtures of Portland cement and organic binders see page 49.

7. *Natural cement*.—In 1903 the production of natural cement in the United States was 7,030,271 barrels, of 300 pounds gross weight each. The average value was \$0.522 per barrel, equivalent to \$3.73 per ton, allowing 20 pounds tare for the barrel.



The tests were made with a sample from Louisville, Ky., which was mixed dry with the coal and then sufficient water was added before pressing. The results were very nearly the same as with Portland cement, the briquets being hard and brittle. In the fire the briquets held together excellently, but in water they would not stand up particularly well. Natural cement would make a cheap binder but would have to be used in such large quantity as to be very objectionable.

8. *Slag cement*.—In 1903, 525,896 barrels of slag cement, of 380 pounds net weight each, worth \$1.03 per barrel, equivalent to \$3.42 per ton, were manufactured in the United States. Tests were made with slag cement as with the other cements, the results indicating its inferiority to either the Portland or the natural cement as a binder for coal slack.

9. *Water glass*.—Water glass, or sodium silicate, is produced to a considerable extent in the United States, 32,651 tons having been manufactured in 1900, with an average value of \$12.74 per ton.

It is said that this material will make coherent briquets when 0.75 to 1 per cent is used. Two different samples were tested. The requisite amount was dissolved in hot water and mixed with the coal, any large excess of water was boiled off, and then the briquets were pressed. The results were unsatisfactory even when 12 per cent of binder was used. The experiments were then repeated with less water and no heat, but the results obtained were no more satisfactory. When the sodium silicate was analyzed one sample was found to contain only 86 per cent of the requisite amount of silica and 13.4 per cent of the requisite amount of sodium required by the formula for the normal silicate ( $\text{Na}_4\text{SiO}_4$ ). The other sample, which behaved only a little better, showed on analysis 11.1 per cent of  $\text{Na}_2\text{O}$  and 27.4 per cent of  $\text{SiO}_2$ . These poor analyses may account to some extent for the lack of success obtained with the water glass, but the results are apparently sufficient to show that it is not suitable for use as a commercial binder.

## ORGANIC BINDERS.

### WOOD PRODUCTS.

10. *Rosin*.—In 1900, 300,000 tons of rosin, valued at \$17.02 per ton, were produced in the United States. Of this amount, according to the Census report, only 7.6 per cent was used for domestic consumption. In 1905 the price of rosin, for even the lower grades, A to C, had risen to \$29 per ton.

Rosin consists mainly of abietic acid or similar isomeric acids or anhydrides. The formula of this acid is given as approximately  $\text{C}_{40}\text{H}_{56}\text{O}_4$ , and its acid equivalent as 145 to 185. This means that if calcium oxide is used to neutralize the acid 0.0725 to 0.0925 gram should be added to 1 gram of the rosin to form calcium resinate.

The density of rosin ranges from about 1.07 to 1.08. Rosin softens at 80° C. and melts to a limpid liquid at 100° C. The melting point of abietic acid is stated to be 165° C. Rosin is entirely soluble in carbon disulphide.

The sample of rosin tested melted at 100° C. The tests made are shown in the table (pp. 51-52). The briquets withstood exposure to the weather well and, except those made with lignites, were satisfactory in the fire, though inclined to smoke.

An attempt was made to see if the addition of lime would improve the binding qualities of the rosin. Three grams of rosin mixed with 0.25 gram of lime melts to a thicker mass, more brittle than the rosin alone. If the amount of lime is increased to 0.50 gram the brittleness is very much increased. Experiments made on Illinois No. 6 B coal, with varying proportions of lime and rosin, gave the following results:

*Results of briquetting Illinois No. 6 B coal<sup>a</sup> with varying proportions of rosin and lime.*

	Percentage of rosin used.			
	2.	4.	6.	8.
First series:				
Lime added.....gram..	0.033	0.067	0.1	0.133
Grade of coherence <sup>b</sup> .....	2	3	3½	3½
Second series:				
Lime added.....gram..	0.066	0.134	0.2	0.266
Grade of coherence.....	2	2½	3	3

<sup>a</sup> Bituminous coal from Coffeen, Montgomery County. For description, analysis, and tests see Bull. U. S. Geol. Survey No. 290, 1906.

<sup>b</sup> See p. 22.

As 20 grams of coal were used the lime added in the first series was just sufficient to react with the rosin. The increase of lime appears from the above results to be detrimental, and the experiments were therefore not carried further. It appears that 6 per cent of rosin will be necessary to produce a satisfactory briquet with most coals, and inasmuch as rosin is now worth about \$29 per ton its use as a binder is unprofitable. Nor is it likely that it will again become cheap enough to permit its use as a binder, either alone or in combination with other materials, such as tar.

11. *Pitch*.—Owing to fluctuations in the price of rosin, pitch, which is a mixture of rosin and tar, is variable in cost. In 1905 a good grade of navy pitch was quoted at about \$35 per ton in St. Louis. The sample of pitch tested was of this grade. For the results of the tests made see table on pages 51-52.

Only 3 or 4 per cent of this pitch is necessary to produce a satisfactory briquet. The briquets stood the weather well and, except those made with the lignites, proved satisfactory in the fire.

The improvement of rosin as a binder by the addition of tar might have been predicted from the principles laid down, for rosin alone is too brittle to produce a tough briquet with a low percentage of binder,



and thinning the rosin with a heavy oil, such as tar, thus making it less brittle, would doubtless be advantageous. However, even where only 3 per cent of pitch is necessary to produce a satisfactory briquet its cost will probably always forbid its use.

12. *Pine-wood tar*.—No accurate data as to the amount of tar produced in the United States could be obtained. The census of 1900 reported 84 wood-distillation plants, but these were mostly using hard woods. The tar produced should be from 4 to 10 per cent of the weight of the hard wood used, but no record of the output was made, the tar being mainly burned under the retorts. The number of distillation plants in the South using pine wood has been considerably increased since the census of 1900, and plants have also been erected to use fir in the northwest. Both pine and fir yield much larger percentages of tar than the hard woods, and it may be that in the future the tar obtainable from these sources will be available for briquetting plants in neighboring sections of the country. The census for 1900 showed exports of 36,535 barrels of tar and pitch, valued at \$77,082, or \$15 per ton. Pine tar is quoted at 6 to 10 cents per gallon, equivalent to \$13.80 to \$23 per ton.

In the distillation of wood various grades of oils and tars are produced, depending both on the wood used and on the manner of distillation. An examination of representative samples of these various grades was undertaken in order to determine their value for briquetting purposes and also to determine how the product could best be made suitable for such purposes.

A solid pine-tar residuum, obtained from Summerville, S. C., was designated 12 A. The final results of the tests made with this binder are shown in the table (pp. 51–52). All the briquets except those made of lignite behaved satisfactorily in the fire. The pitch softened at 80° to 90° C. to a very sticky mass that apparently should bind well, but some of the briquets, even with 10 and 12 per cent of the binder, were too brittle, although they were sufficiently hard. The poor results with this binder were attributed to the high percentage of carbon in the pitch and to the failure of the pitch to spread well over the grains of coal. The pitch dissolved readily in either wood-tar creosote or coal-tar creosote. The following tests were made:

*Results of briquetting Arkansas and Illinois coals with varying proportions of pine-wood tar and creosote oil.*

Coal.	Binder (per cent).			Grade of coherence. <sup>a</sup>
	Pine-wood tar 12 A.	Wood creosote oil.	Coal-tar creosote oil.	
Arkansas A.....	5	2	.....	4½
	5	.....	2	4½
Illinois No. 11 B.....	6	2	.....	4
	6	.....	2	4

<sup>a</sup> See p. 22.

As was to be expected, these briquets smoke, but they stand up satisfactorily in the fire. The experiments show the improvement which may be made by thinning a pitch to the proper consistency. This holds also for coal-tar pitches, as will be seen later.

The pitch here discussed is a waste product, but being produced at only a few plants is not available in quantity.

A sample of very thick pine-wood tar, obtained from Cheraw, S. C., was designated 12 B. Its flowing point was 45° C. and only 3 per cent was volatile below 270° C., the volatile portion being mostly water. This tar had a density of 1.07. The results of the experiments made with it are summarized in the table (pp. 51-52).

The briquets produced some smoke, but were satisfactory in the fire except when made with lignite. They stood the weather well. This tar may prove an available binder for some briquet plants. It is obtainable at many wood-distillation plants at prices ranging from \$15 to \$20 per ton, and as only 3 to 4 per cent is necessary to produce a satisfactory briquet with most coals the binder would range in price from 45 to 80 cents per ton of briquets produced.

Another sample, of a slightly more mobile tar than 12 B, obtained from the same plant, was designated 12 C. Its flowing point was 42° C. and its density 1.05. About 14 per cent of this tar distilled below 270° C. The results of the experiments with it are given in the table (pp. 51-52). This tar is obtainable from any of the wood-distillation plants that could furnish tar like the sample 12 B, and would command about the same price. It contains a little more of the low-boiling oils—that is, those distilling below 270° C.—than sample 12 B, and requires about 1 per cent more of the tar to produce a satisfactory briquet.

A sample of pine tar obtained at St. Louis, Mo., was designated 12 D. It was liquid at 20° C. and had a density of 1.14. On distillation about 10 per cent came off below 200° C. and 25 per cent below 270° C. The following experiments were tried:

*Results of briquetting Illinois No. 6 B coal with binder 12 D.*

Percentage of binder.	Grade of coherence. <sup>a</sup>
2	3
4	3
6	3
8	3
12	4

<sup>a</sup> See p. 22.

The tar was evidently too liquid to produce satisfactory briquets. The residue left after distillation at 270° C. was then tested and gave a satisfactory briquet with Illinois No. 6 B coal when only 4 per cent of binder was used.

Another sample of pine-wood tar, obtained from a wood-distillation plant at Dunbar, S. C., was designated 12 E. It was found that about 5 per cent of this tar would produce a satisfactory briquet with Illinois No. 6 B coal.

Another sample of pine-wood creosote, obtained from Cheraw, S. C., was designated 12 F. This sample was liquid at 20° C. and had a density of 1.12. On distillation about 20 per cent by volume came off below 112° C., the distillate being mostly water, and 21 per cent more came off below 270° C. At 310° C. the residue swelled up and frothed over. The briquets made with this binder were not satisfactory, the reason being that the creosote was so thin that the briquets were easily crushed. They smoked in the fire, gave off the odor of creosote, and did not stand up well. The residuum left after the distillation of the creosote had been carried to 270° C. was tested with Illinois No. 6 B coal, the coherence being 3 and 4 with 6 and 8 per cent of binder, respectively.

A sample of pine-wood creosote, obtained from a plant at Dunbar, S. C., designated 12 G, was not tested, being similar to 12 F, with which no satisfactory results could be obtained. Another sample of turpentine oil obtained from the same plant, designated 12 H, was evidently of no value for briquetting purposes.

13. *Hard-wood tar*.—The sample of hard-wood tar examined was a rather thin liquid even at the ordinary temperature, and could not therefore make a sufficiently hard briquet. It had a density of 1.10. The following tests were made:

*Results of briquetting Illinois No. 6 B coal with hard-wood tar.*

Percent- age of binder.	Grade of coher- ence. <sup>a</sup>
2	2
4	2
6	3
8	3
12	3½

<sup>a</sup> See p. 22.

On distillation below 112° C. the tar gave off 8 per cent of water; from 112° to 270° C. it yielded 44 per cent more of a light oil and of reddish paraffin oils. On testing the residue a satisfactory briquet was obtained with Illinois No. 6 B coal when 8 per cent was used as a binder. It is concluded, therefore, that the residue left from hard-wood tar after distillation to 270° C., where it is obtainable, could be used advantageously for briquetting.

14. *Fir tar*.—The sample of fir tar tested was obtained from a wood-distillation plant in the State of Washington. On distillation the tar gave off 8 per cent below 270° C. The results of the tests



are shown in the table (p. 51). As will be seen, the tar produces satisfactory briquets when 6 per cent is used.

Concerning the use of wood tar in general for briquetting, the conclusions to be drawn are that the distillation of the tar should in general be carried to 270° C., and the residue, which will be either a thick tar or a soft pitch, should be used. The briquetting qualities of a tar thus prepared will vary considerably with the source of the tar. Pine tar is best, about 4 per cent being required; fir tar comes next, about 6 per cent being required; and lastly, hard-wood tar, about 8 per cent being required to produce a satisfactory briquet. The work has not been extended to a sufficient number of samples of tar to make the above conclusions as regards the percentage of each tar required absolutely certain, but the percentages given will serve as the basis for a rough estimate of the cost of wood tar as a binder. In some localities this product might compete successfully with other binders.

15. *Wood pulp*.—The claim has been made that cellulose, which is the main constituent of prepared wood pulp, has binding properties, but a few experiments point to the conclusion that its use is wholly impracticable. Possibly the term was confused with lignocellulose, the lignone groups affording the main constituents of the sulphite liquor discussed in the next section.

16. *Sulphite liquor*.—In the manufacture of paper, wood pulp is treated with sulphurous acid to remove certain lignone groups, which combine with the  $\text{SO}_3\text{H}$  and are then removed in the waste water, in which they are soluble. This waste liquor, amounting to ten or twelve times as much as the cellulose fiber produced, yields on evaporation an average of 9 to 10 per cent of solid residues. Roughly, therefore, the amount of this solid waste material is equal to the amount of cellulose obtained. According to the United States Census report for 1900 the amount of sulphite fiber produced was 416,037 tons, and the estimate indicates that there was an equal production of the waste lignone complex.

Not only is this liquor a true waste material, finding at present no market, but its production is a great nuisance, for it very seriously pollutes the streams on which the mills are situated and gives rise to much trouble. Its cost, therefore, would be represented solely by the cost of getting rid of the excess of water and by the freight to the briquetting plant. The water could be removed by evaporation, during which process the complex groups are to some extent broken down, sulphur and sulphur compounds being formed and some of them escaping. Of the solid residue left from evaporation 20 per cent is inorganic material and 80 per cent is organic. An ultimate

analysis of the lignone complex groups shows, according to Cross and Bevan, carbon, 50.22 to 56.27 per cent; hydrogen, 5.22 to 5.87 per cent; sulphur, 5.52 to 8.80 per cent.

Efforts have been made by various investigators to separate the lignone complex groups from the water by precipitation instead of by evaporation. It is possible that some process of settling and filtration may recover the desired gummy residues without going to the expense of evaporating the water, but the processes so far devised are as yet unsuccessful on a commercial scale.

The sample of sulphite liquor examined was obtained from Detroit, Mich. On evaporation it showed a dry residue of 11.8 per cent. This residue, of course, does not melt but chars and decomposes if heated to a high temperature. Before evaporating quite to dryness the residue is a very sticky, gummy mass, easily soluble again in water. The original liquor was evaporated to about one-third of its bulk and when in this condition was used in the following tests:

*Results of briquetting California lignite<sup>a</sup> and Illinois coal with sulphite liquor.*

Percent- age of binder.	Grade of coherence. <sup>b</sup>	
	Califor- nia No. 1.	Illinois No. 6 B.
2	.....	2
4	.....	2½
6	.....	3
8	.....	3½
10	3½	4
12	4	4½

<sup>a</sup> Lignite from Tesla, Alameda County. For description, analysis, and tests see Bull. U. S. Geol. Survey No. 290, 1906.

<sup>b</sup> See p. 22.

The briquets from Illinois No. 6 B coal, with 10 per cent binder, and from California No. 1, with either 10 or 12 per cent binder, were satisfactory in the fire. The briquets made from the California lignite show the good effect of using a binder which does not volatilize or melt, for this coal is one of the most difficult of all the coals with which to obtain satisfactory results in the fire. In water, of course, the briquets will go to pieces rapidly.

It must be remembered that the above percentages of binder refer not to the dry residue from the sulphite liquor, but to the liquor itself when concentrated only to one-third of its bulk. To compare the results with the dry material the percentages must be divided by three. In other words, we have from the paper mills each year 1,200,000 tons of waste material which will produce coherent briquets when 10 to 12 per cent of it is used as a binder. The drawback to its use is the fact that the briquets are not waterproof, and a few

preliminary experiments were made in an endeavor to overcome this difficulty, with the following results:

*Results of briquetting California lignite and Illinois coal with varying proportions of sulphite liquor and other binders.*

Coal.	Binder (per cent).			Grade of coher- ence, <sup>a</sup>	Fire test.	Water test.
	Sul- phite liquor.	Waterproofing constituent.				
		Material.	Amount.			
California No. 1 (lignite) .....	{ 8	Pitch 39.....	4	4	Fair....	
	{ 8	do.....	8	4	O. K....	Fair.
	{ 6	Coal-tar creosote.....	4	3½	O. K....	
Illinois No. 6 B.....	{ 6	Asphalt tar.....	4	4	O. K....	Fair.
	{ 6	Pitch 39.....	4	4	O. K....	Fair.

<sup>a</sup> See p. 22.

These experiments indicate that oils and pitches mixed with the sulphite liquor will render the briquet more or less waterproof, depending on the extent and character of the added constituent. The whole problem is an important and promising one and deserves further investigation.

#### SUGAR-FACTORY RESIDUES.

17. *Beet pulp*.—Several samples of beet pulp (a waste product) were examined and carefully tested in the hope that they might contain sufficient starchy or sugary material to serve as a binder. The results showed that the pulp could be of no use whatever for this purpose. Details of the tests need not therefore be given.

18. *Lime cake*.—The sample of lime cake examined proved to be practically pure calcium carbonate, which could be of no possible use in briquetting.

19 and 20. *Beet-sugar molasses and cane-sugar molasses*.—The binding power of molasses is said to be due to pectin, which is a body closely related to mucilage and has the constitution of a typical lignocellulose. To a less extent the binding power is due to sugar. Molasses contains only about 10 per cent of ash. From 1 to 1.5 per cent of molasses in water is said to be sufficient for binding, but the experiments do not verify the statement. Three samples of beet-sugar molasses were examined—19 A, 19 B, and 19 C. Samples 20 A and 20 B were cane-sugar molasses. The moisture and ash were determined as follows:

*Moisture and ash in beet-sugar and cane-sugar molasses.*

	Beet-sugar samples.			Cane-sugar samples.	
	19 A.	19 B.	19 C.	20 A.	20 B.
Moisture.....per cent..	12.5	13.8	21.8	27.3	27.5
Ash.....do.....	8.1	10.1	9.9	6.3	5.7



Tests in briquetting Illinois No. 6 B coal with these samples gave the following results:

*Results of briquetting Illinois No. 6 B coal with varying percentages of beet-sugar and cane-sugar molasses.*

Percentage of binder.	Grade of coherence. <sup>a</sup>				
	19 A.	19 B.	19 C.	20 A.	20 B.
2	3	2			
4	3	3	2	2½	2
6	3	3	3	3	3
8	3	3	3	3	3
12	3	3	3	3	3

<sup>a</sup> See p. 22.

The coherence of the briquets did not seem to be increased by using more than 6 per cent of molasses. The failure to obtain good briquets with smaller percentages or to obtain satisfactory briquets even when the higher percentages were used is hard to explain. Heating the briquets to a higher temperature, even to 150° or 160° C., did not seem to improve them. Their behavior in the fire could not be regarded as very satisfactory. In water they fell to pieces.

Some experiments were made with lime and molasses and also some attempts to waterproof these briquets, but no very satisfactory results were obtained. The use of molasses as a binder needs further investigation before it is finally classed as being of no use for briquetting, but so far it would seem to be without commercial value for this purpose.

The census report for 1900 showed that there were 3,551,856 gallons of this molasses produced, valued at \$25,102 for the portion sold. Much of it went to waste.

#### STARCH.

21. *Cornstarch*.—In the tests of cornstarch it was first necessary to determine if heating the starch with water to a paste, thus forming dextrin, before mixing it with the coal was essential, or if the change of starch into dextrin would take place as well when the starch was first mixed with the coal and the mixture then moistened and heated. The experiments showed that the latter procedure was fully as effective. Starch was tested more particularly with the lignites, because it does not evaporate before burning, and hence would hold the lignite together in the fire. The results of the tests are shown in the table (pp. 51-52). In all the tests the behavior of the briquets in the fire was far more satisfactory than if pitch or a similar binder had been used. Starch possesses the advantage over such binders that it adds no smoke-producing material to the coal.

In water these small starch briquets fell to pieces in a few minutes, and the next endeavor was to waterproof them. Many attempts were

made to accomplish this end by immersion in oil. The experiments indicated that any oil would waterproof the briquet when externally applied, but asphalt tar, which was the thickest oil tested, gave the best results. It is doubtful if external waterproofing with a thick oil would ever be commercially successful, owing to the cost and difficulty of manipulation, but a thin oil, such as crude petroleum, might answer. At any rate, laboratory tests with small briquets can not finally decide the point, and the experiments should be conducted on a larger scale.

An endeavor was also made to waterproof by mixing the coal and starch with some of the oils before briquetting. For this purpose Hoffman's petroleum, Kansas crude oil, coal-tar creosote, asphalt tar, water-gas tar pitch, coal-tar pitch, and hard-wood tar were used under varying conditions and with varying percentages both of the starch and of the oils. The experiments indicate that the presence of crude oil or tarry liquids is detrimental to the action of the starch, both as to coherence and in the fire. But the binding power of the starch, though somewhat diminished, was nevertheless still very great, and it is probable that a briquet with 1 per cent of starch and 8 per cent of a heavy crude oil, or a less percentage of oil residue, would prove satisfactory. It is possible that in some places such a combination might prove the cheapest and most satisfactory binder obtainable. Pitches did not seem to injure the action of the starch, but unless a small percentage of pitch is found to waterproof there would be nothing gained by the combination. The experiments made did not seem to indicate that a small percentage of pitch with starch would give satisfactory results in the weather, but this point should be tested on a larger scale.

A patent for the use of starch as a binder was issued in 1858, in England, to John Piddington. He used 36 pounds of starch and 8 per cent of water per ton of coal.

The objections to starch as a binder are that the briquets do not immediately harden, and that they will not stand exposure to the weather unless made waterproof. The advantages of starch as a binder are its cheapness, its wide availability, the fact that it introduces no smoke, and the fact that, being nonvolatile, it holds the coal together well.

As shown by the census report for 1900 the amount of starch produced in the United States during that year was 297,803,139 pounds. Of this amount 247,051,744 pounds was made from corn as raw material, the average price of the starch being 2.5 cents per pound. It is of course not necessary that starch to be used as a binder be pure, and a far better idea of its cost for this purpose can be obtained by considering the cost of the raw material.

The raw materials available in the United States are corn, wheat and other small grains, Irish potatoes, sweet potatoes, cassava, and spoiled products containing starch. The starch from wheat and other small grains is more expensive than that from corn. Cassava, yielding 4 to 5 tons per acre and containing about 25 per cent of starch, offers a very cheap source of starch, but in the United States it can not be grown far north of Florida.

In 1900, 231,106 tons of corn were used for the production of corn-starch, the average price paid being \$11.78 per ton. Corn contains 60 to 65 per cent of starch. The factories extracted on the average 53.4 per cent, and the cost of this starch in the crude condition is therefore \$18.85 per ton. The only preparation necessary would be fine grinding.

The price of raw cornstarch may be estimated at \$20 per ton, based on the census report for 1900, and inasmuch as only 0.5 to 1 per cent of this material is required to make a coherent briquet, it follows that the cost of starch binder of this kind per ton would be only 10 to 20 cents. The briquets would not stand rain, but would prove perfect if kept under cover. It seems that starch briquets, only slightly waterproofed, might be used during the dry season in certain sections of the West. If more thoroughly waterproofed with heavy crude petroleum oils they might be generally used. The crude petroleum would increase the fuel value of the briquet almost sufficiently to pay for itself. It seems, therefore, that further experiments with starch on a larger scale are desirable.

22. *Potato starch*.—Properly chosen varieties of the sweet potato contain about 22 per cent of starch and the yield per acre is large. Small, unmarketable potatoes may be used. The sweet potato is available in many parts of the United States.

The Irish potato is widely distributed, and starch factories consumed 118,000 tons in 1900, paying an average of \$5.90 per ton and obtaining an average of 14.3 per cent of starch. As a rule only unmarketable potatoes were used and this accounts for the low percentage of starch obtained, the average yield of Irish potatoes being 18.2 per cent of starch, and some varieties giving as high as 25 per cent. On the basis of 18 per cent available starch, the raw starch obtained from this source is worth \$32.75 per ton.

Usually, therefore, starch obtained from potatoes would be more expensive than that obtained from corn. A number of tests were made to see if the action of the two starches is similar. No difference in the coherence of the briquet or in its behavior in the water or in the fire was detected.



## SLAUGHTER-HOUSE REFUSE.

Slaughter-house refuse, which is now largely made into glue, has been so often suggested as a binder that its cost was investigated. The census of 1900 showed that 34,750 tons of glue were produced, valued at \$155 per ton. The price is therefore prohibitive and no experiments were made with this material.

## TARS AND PITCHES FROM COAL.

*Preliminary considerations.*—The work done at the briquetting plant under the direction of Dr. J. H. Pratt had shown that there was great variation in the value of various coal-tar pitches for briquetting purposes. That work had also shown that coal-tar pitch would be one of the most important binders to be considered. An endeavor was made, therefore, to study the various grades of coal tar and the pitches therefrom, with the idea of improving the pitches and of establishing some method of examination which might reveal their value without the necessity of an actual briquetting trial.

The total production of coal tar in the United States in 1903 was 62,964,393 gallons, valued at \$0.0349 per gallon, or \$7.27 per ton.

On distillation coal tar is divided into several fractions which are more or less clearly defined. By further distillation these fractions are separated more completely and find their way to the market as illuminating oils, naphtha, creosote, etc. They consist of a very large number of chemical compounds. The manner in which coal tar is fractionated varies at different works, but as illustrative, it may be said that the ammoniacal liquor distils first, then the first light oils, boiling below 110° C. The second light oils come off at 110° to 170° C., the carbolic oils at 170° to 225°, the creosote oils at 225° to 270°, the anthracene oils at 270° to 360°, and lastly the pitch is left behind as a residue.

None of the oils coming off below 270° C. are useful in briquetting. The anthracene oils, which consist of a large number of different compounds, should not, however, be entirely distilled from the pitch if it is desired to use the pitch for briquetting. Nearly all the various constituents of both the pitch and the anthracene oils except the free carbon are soluble in carbon disulphide. Constam and Rougeot<sup>a</sup> examined 33 pitches obtained from various sources, and found the amount of carbon-disulphide extract to range from 60.43 to 91.22 per cent and to average 76.3 per cent. They also found the value of the pitch for briquetting purposes to be proportional to the amount of carbon-disulphide extract. The results obtained by the writer lead to the same conclusion, except that the free carbon (that is, the insoluble portion) is believed to be not only inert but detrimental to

<sup>a</sup> Zeitschr. f. angew. Chemie, vol. 17, no. 26.

the pitch, indicating that the increase in the value of a pitch for briquetting purposes is somewhat greater proportionally than the increase in the percentage of extract obtainable. The free carbon seems to prevent the pitch from spreading easily over the grains of coal, and owing to its very finely divided condition itself offers a very large surface for the absorption of pitch.

A pitch has no true melting point, but owing to the large number of different chemical bodies which it contains, softens only very gradually. This softening point of the pitch has a marked influence on its use in briquetting, for the pitch must either be so brittle that it can be broken finely and mixed with the coal as a solid, or it must be melted and distributed as a liquid. Many pitches soften at so high a temperature that they can not be efficiently used except by heating above 100° C. The pitch must therefore be adapted to the briquetting machine in which it is to be used. Many methods of determining the softening point of a pitch have been suggested, but most of them are either too troublesome for practical use or not accurate. In the experiments here recorded the flowing point of the pitch was used as an index of the temperature at which it softened. This point was determined by placing about 3 cubic centimeters in the bottom of a test tube one-half inch in diameter and inserting the tube in a bath. The temperature of the bath was raised until, on taking out the tube and inverting it, the pitch flowed 1 inch down the tube in fifteen seconds.

In ascertaining the value or suitability of a given pitch or tar for briquetting purposes three determinations are necessary:

1. The pitch or tar is distilled and all oils coming off below 270° C. are rejected as being of no value in briquetting.
2. The flowing point of the portion to be used in briquetting is determined. This should generally be not less than 70° C.
3. The pitch is extracted with carbon disulphide. The smaller the amount of residual carbon the more satisfactory the pitch.

It should be borne in mind that the higher the flowing point of the pitch the more satisfactory it will prove in the fire when used with coals that do not cake readily. If the pitch has too high a flowing point to be workable with the briquet machine at hand, it could be softened by the addition of a high-boiling coal-tar oil (above 270° C.) or of very soft pitch. Coal-tar creosote could be used, but its boiling point is too low to make its use in all respects satisfactory.

23. *Blast-furnace tar*.—As it was impossible to learn whether blast-furnace tar and the similar material known as shale tar are produced in the United States, no experiments were made with them.

24. *Producer-gas tar*.—Two samples of producer-gas tar were examined. The first, designated 24 C, after pouring off the water, gave on distillation, water, 30 per cent; oils below 270° C., none;

oils at 270 to 330° C., 6 per cent. From 330° the thermometer jumped suddenly to 370° and the distillation was stopped. The residue gave with Illinois No. 4 coal a satisfactory briquet when only 4 per cent was used as a binder.

The next sample, designated 24 D, was tested after boiling off the water. The result showed a satisfactory briquet with Arkansas No. 7 A coal when 4 per cent was used, but a larger percentage is necessary with most other coals and probably 8 per cent would be necessary for most lignites.

The tar obtained, when freed from water only, is rather too liquid to produce the best quality of briquet. But the removal of only about 6 per cent of oils raises the flowing point of the tar to about 70° C. and the residue appears, as above seen, to be excellently fitted for briquetting purposes. The amount of carbon-disulphide extract obtainable from the residue was not determined. It should not be large, for the temperature at which the tar is made is comparatively low. This is probably the cause of the superior binding power of the pitch.

The amount of this tar obtainable and its market value are questions for future determination.

25. *Illuminating-gas tar*.—About 25 per cent of the illuminating gas produced in the United States is made from coal, and the tar resulting from the process amounts to about 5 per cent of the coal coked. The census report for 1900 gives the production for 1899 as 67,094 tons. In 1903, 61.4 per cent of the coal tar made was produced in gas works. The average value of this tar as distinct from other coal tars is not obtainable, and \$7.27 per ton, the average value of all coal tars for 1903, is therefore accepted as approximately correct for gas tar.

This tar is too liquid to produce good briquets. The oils coming off below 270° C. should be disposed of. The residue, equaling 70 per cent of the total, would cost \$10.40 per ton, if the sale of the low-boiling oils could be made to pay the expense of the distillation and the profit thereon.

Pitches 28 A, 28 B, 28 C, 28 D, 28 E, 28 F, and 28 I, obtained from this tar, were examined, and the percentages determined as necessary to make satisfactory briquets are shown in the table (pp. 51-52).

26. *By-product coke-oven tar*.—In 1903, 38.6 per cent of the total coal tar produced (24,296,536 gallons) was produced in by-product coke ovens. The census report for 1900 shows that in 1899 only 3.33 per cent of the total coal coked was coked in by-product ovens. Consequently the amount of coal tar from this source could be enormously increased.

This tar is obtained by distillation at a high temperature, and therefore contains more fixed carbon than tar from illuminating-gas



plants. About 60 per cent of the tar from an Otto-Hoffman oven is pitch.

The tar is too liquid to be used directly for briquetting. The results with pitches 28 G and 28 H, made from coke-oven tar, are shown in the general table (p. 51).

27. *Coal-tar creosote*.—The principles governing the use of binders make it appear useless to test coal-tar creosote alone. It is too thin a liquid to make coherent briquets and of too low a boiling point to give satisfactory results in the fire. This creosote could be used to thin a pitch whose boiling point is too high, when such use is advantageous. It could also be used to waterproof a binder that would not stand the weather; but this could be done as well with a crude oil of low specific gravity, and the cost would be less. Coal-tar creosote is worth about 6 cents per gallon.

28. *Coal-tar pitches*.—The pitch designated 28 A was obtained by Dr. J. H. Pratt, who called it pitch C in his report.<sup>a</sup> This pitch flowed at 100° C., and the behavior of the briquets in the fire was satisfactory.

The pitch designated 28 B was obtained through Dr. Pratt and was by him designated, in his report, pitch D. This pitch had a flowing point of 127° C. It was used in making a very large number of tests on the comparative action of different coals with the same binder.

The pitch designated 28 C was used to briquet 50 tons of Arkansas semianthracite slack, about 6.5 per cent being used. This pitch had a flowing point of 100° C. It proved too soft for use with a pitch cracker on a summer day.

The pitch designated 28 D was used to briquet 200 tons of Arkansas semianthracite slack, about 6.5 per cent being used. This pitch had a flowing point of 120° C. and was sufficiently brittle for use on the hottest day. It gave a carbon-disulphide extract of 67.75 per cent.

The pitch designated 28 E had a flowing point of 100° C.

The pitch designated 28 F was very soft, having a flowing point of 68° C., and did not prove as efficient a binder as its appearance indicated. No further examination was made to determine the cause of the trouble.

The pitch designated 28 G was a soft coke-oven pitch obtained from tar produced in the Semet-Solvay process. It had a flowing point of 95° C. and yielded about 86 per cent of carbon-disulphide extract.

The pitch designated 28 H was a harder coke-oven pitch from the same source as 28 G. It had a flowing point of 100° C. and gave 81.50 per cent of carbon-disulphide extract. The briquets were possibly a little stronger than those made with the soft coke-oven pitch.

<sup>a</sup> Preliminary report on the operations of the coal-testing plant of the United States Geological Survey at the Louisiana Purchase Exposition, St. Louis, Mo., 1904; Bull. U. S. Geol. Survey No. 261, 1905, p. 134.

The pitch designated 28 I was received through Dr. Pratt, who called it pitch X in his report.<sup>a</sup> It had a flowing point of 190° C., being very hard and brittle. The carbon-disulphide extract was 63.2 per cent. A large number of tests were made with this pitch when determining the qualities of binders in general in order to learn why this grade was so poor a binder. This seemed to be due to two causes—(1) the large amount of contained free carbon (36.8 per cent), and (2) the high softening point. At 100° C. the binder did not melt sufficiently to spread over the grains of coal to the best advantage.

To test this latter point the pitch was mixed with wood creosote 12 F, which did not itself possess sufficient binding power. Two hundred grams of pitch was mixed with 100 grams of the wood creosote and heated with stirring until thoroughly mixed. The resultant pitch, which was brittle enough to be pulverized if kept cool, was then tried with a number of coals and compared with the original pitch. The results from this mixture, designated 28 J, are shown in the table (pp. 51–52). It will be noted that in all the tests 4 per cent more of the original pitch than of the mixture was required, thus confirming the diagnosis of the trouble.

None of the coal-tar pitches gave coherent briquets with less than 6 per cent, and with many of them 7 or 8 per cent was required. The reason why a coal-tar pitch will not briquet if less than 6 per cent is used is that it contains a comparatively large amount of carbon. The residue from producer-gas tar made satisfactory briquets with 4 per cent, and this result was doubtless due to the fact that such tar contains little free carbon.

The cost of coal-tar pitch per ton may be taken as \$11; therefore the cost of the binder per ton of briquets produced ranges from 66 to 88 cents. The briquets when properly made will stand exposure to the weather well. They will stand up satisfactorily in the fire if the coals cake at all readily. With noncaking coals the briquets would not prove satisfactory in the fire. This binder does not cause an undue amount of smoke.

#### NATURAL ASPHALTS.

Asphalts grade almost imperceptibly into heavy, thick petroleum oils. The designations used by Eldridge<sup>b</sup> have been followed in this discussion. Wurtzilite, nigrite, ozocerite, and grahamite occur in the United States, but not in deposits profitable to mine.

<sup>a</sup> Preliminary report on the operations of the coal-testing plant of the United States Geological Survey at the Louisiana Purchase Exposition, St. Louis, Mo., 1904; Bull. U. S. Geol. Survey No. 261, 1905, p. 134.

<sup>b</sup> Eldridge, G. H., Origin and distribution of asphalt and bituminous rock deposits in the United States: Bull. U. S. Geol. Survey No. 213, 1903, pp. 296–305.

30. *Impsonite*.—Impsonite, sometimes called grahamite, is found in Oklahoma. It softens at a high temperature, but does not melt. In carbon disulphide 35 per cent or more is dissolved.

The sample tested was obtained through Dr. Pratt and was designated B 4 in his report.<sup>a</sup> This was tested with a lignite, as its only possible use in briquetting was considered to be to mix with a non-caking coal in rather large percentage. From 20 to 30 per cent was found to be required to hold a California lignite together in the fire. Even though the material is very cheap, the large percentage required prohibits its commercial use.

31. *Gilsonite*.—It is estimated by Eldridge that 32,000,000 tons of the asphalt known as gilsonite are now in sight in the extensive deposits that occur in Utah. He further states that the cost to mine does not exceed \$1.75 per ton. The material has to be hauled a long distance to a railroad, and the present price in St. Louis is about \$35 per ton. Gilsonite has a brilliant luster, burns and acts like sealing wax, and is entirely soluble in carbon disulphide. Two samples were tested.

The sample designated 31 A was black, with a brilliant luster, and flowed at about 250° C. In testing, the finely powdered material was mixed dry with the coal and heated far above 100° C. As shown in the table (p. 52) it gave a good briquet when 4 per cent was used.

The sample designated 31 B was black, with a less brilliant luster. When its flowing point was being determined it frothed out of the tube. It gave a briquet of satisfactory coherence when 6 per cent was used as a binder. The briquets are also satisfactory in the fire; and, owing to the high softening point of the binder, it would be very useful with noncaking coals. At its present price of \$35 per ton, however, even 4 per cent of this binder is out of the question.

32. *Maltha*.—Small deposits of maltha, a liquid asphalt, occur in Oklahoma, Mexico, California, and Texas. In 1903 the only production reported to the Geological Survey was 58 tons from Texas, valued at \$19.83 per ton.

The sample tested was obtained through Dr. Pratt and was called by him "liquid Austin asphalt." A satisfactory briquet was produced with 3 to 3½ per cent of binder. Attention is called to the fact that when as much as 8 per cent of this binder is used the briquet grows weaker instead of stronger. This is due to the low flowing point of maltha, 58° C., which causes the briquet to crush easily if an excess is used. In the fire the binder would give satisfactory results only when used with coals that cake very easily.

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<sup>a</sup> Preliminary report on the operations of the coal-testing plant of the United States Geological Survey at the Louisiana Purchase Exposition, St. Louis, Mo., 1904; Bull. U. S. Geol. Survey No. 261, 1905, p. 134.



The cost of this binder, 3 per cent being used, would be 60 cents per ton of briquets produced. With some coals a larger percentage would be necessary.

33 and 34. *Refined Trinidad asphalt and refined Bermudez asphalt.*—Considerable quantities of crude Trinidad and Bermudez asphalts are annually imported. In 1903 the imports of the former amounted to 129,133 tons, valued at \$367,003; and of the latter 9,898 tons, valued at \$48,218.

The cans in which samples were furnished for these experiments were not marked and complete identification was impossible. The softer of the two samples flowed at 115° C. and could not be powdered. It gave a satisfactory briquet when 6 per cent was used with Illinois No. 11 C (4) coal. The harder sample could be powdered, flowed at 180° C., and on testing showed a briquet that was hardly satisfactory when 8 per cent of the binder was used with Illinois No. 11 C (4) coal. If the binder had been superheated better results could probably have been obtained.

These asphalts apparently could not compete with coal-tar pitches as binders.

35. *Hard and refined asphalts.*—Bituminous sandstones, limestones, or shales occur in several States in deposits of considerable extent. These are mined, but usually the rock is used as a constituent of paving mixtures and the bitumen is not extracted. Attempts have been made to refine this rock either by distillation or by extracting the bitumen with a solvent, such as naphtha. The process does not seem to have been very successful commercially. The only production reported is 6,400 tons from California, with a value per ton of \$21.87; and 877 tons from Indian Territory, with a value per ton of \$17.61. No samples could be obtained, and the product is probably not now on the market.

#### PETROLEUM PRODUCTS.

36. *Crude oil.*—Unless they are of the consistency of maltha, crude oils are not suitable for binders, being too liquid. They might be used to advantage in waterproofing briquets made with starch, sulphite liquor, or molasses.

37. *Petroleum residuum.*—There are many grades of petroleum residuum depending on the base of the crude oil (that is, whether the oil has an asphalt base, or a paraffin base, or an asphalt and paraffin base), on the temperature at which the distillation is stopped, and on the amount of cracking to which the oil is subjected during the distillation.

In 1903, 46,000 tons of asphaltic residue, with an average value of \$11.30 per ton, were produced from petroleum in California; and

2,100 tons, valued at \$14.16 per ton, were produced in Texas. If 4 per cent of this material were used as a binder, the cost per ton of briquets produced would be 45 to 55 cents per ton, making this binder one of the cheapest to be had near the oil fields, when the oil contains an asphalt base. Even less than 4 per cent could be used with some coals. For the best results, the asphalt residue should flow at 90° to 100° C.

Six samples of asphalts were examined. The sample designated 37 A was shown by test to flow at 100° C., and 99.38 per cent was soluble in carbon disulphide. The tests showed that except with the lignites, 3 to 4 per cent of this asphalt would give a satisfactory briquet. With caking coals it is satisfactory in the fire.

Another sample was designated 37 B. With most coals 3 to 4 per cent of this asphalt would be required to produce satisfactory briquets.

The sample designated 37 C was received through Dr. Pratt from Caspar, Wyo., and by him was designated B 6 in his report.<sup>a</sup> It flowed at 95° C., and gave a carbon-disulphide extract of 99.88 per cent. A satisfactory briquet was made with 4 per cent of this binder.

The sample designated 37 D was received from Texas, and was designated B 3 in Dr. Pratt's report.<sup>a</sup> It flowed at 140° C., and with most coals about 6 per cent would be required to produce a satisfactory briquet.

The sample designated 37 E, a California asphalt of grade B, was designated B 1 by Dr. Pratt.<sup>a</sup> It did not soften sufficiently at 100° C., but if superheated a satisfactory briquet could be obtained with 8 per cent as binder.

The sample designated 37 F, a Texas asphalt, was designated B 2 by Dr. Pratt.<sup>a</sup> It did not soften sufficiently at 100° C. When superheated it gave a satisfactory briquet with Illinois No. 4 coal, 6 per cent of binder being used.

38. *Water-gas tar*.—The census report states that 75 per cent of the illuminating gas produced in the United States in 1899 was water gas. Petroleum oil is used in enriching this gas and is partly decomposed in the process, resulting in the formation of water-gas tar, of which 48,714,324 gallons were produced in 1899. With an average density of 1.1, this would be equivalent to 222,868 tons of tar. The tar itself is too liquid for use, but a pitch made from it was examined, as shown in the next paragraph.

39. *Water-gas tar pitch*.—The sample of water-gas tar pitch furnished to Dr. Pratt was by him designated pitch H.<sup>a</sup> It flows at 92° C., and with some of the coals 5 per cent proved sufficient to produce excellent briquets. The carbon-disulphide extract was 88.10 per cent. With caking coals the briquets are satisfactory in the fire. This pitch is worth somewhat less than coal-tar pitch, its value being

<sup>a</sup> Preliminary report on the operations of the coal-testing plant of the United States Geological Survey at the Louisiana Purchase Exposition, St. Louis, Mo., 1904; Bull. U. S. Geol. Survey No. 261, 1905, p. 134.

given approximately as \$10 per ton. The cost of the binder per ton of briquets produced would therefore be about 50 cents, effecting a saving of at least 20 cents per ton over the use of ordinary coal-tar pitch.

40. *Wax tailings*.—A product known as wax tailings was received by Dr. Pratt. It is soft at ordinary temperatures and pulls into long threads. It melts to a thin liquid at about 70° C. As low as 3 per cent gives briquets of satisfactory coherence and these are also satisfactory in the fire if the coal cakes readily. The briquets could not be subjected to any pressure in the fire, and would yield to pressure if placed in a warm place. It is doubtful if they could be piled in a very hot sun. The yield of this product is said to be moderate in amount. The value is 6 cents per gallon, or about \$15 per ton, and the cost of this binder would therefore be 45 to 60 cents per ton of briquets produced.

41. *Acid sludge*.—Tests of a sample of acid sludge showed that 10 to 12 per cent was necessary to make a coherent briquet. This material was distinctly acid with sulphuric acid. Its value could not be learned and therefore no further experiments were tried with it.

42. *Asphalt tar*.—The product known as asphalt tar, as obtained by Dr. Pratt, was a rather thin liquid which poured readily and produced briquets that would crush easily and would not stand up satisfactorily in the fire. This tar, if its price permitted, might be used for waterproofing briquets made with soluble binders, as starch, sulphite liquor, or molasses.

43. *Pintsch gas tar*.—Pintsch gas tar, produced by the heating of petroleum oil in iron retorts to a high temperature, is obtained as a thin emulsion in water, being too thin for use as a binder. As it is produced only in very small amounts in the United States, its further examination was deemed inadvisable.

44. *Pittsburg flux*.—The substance known as Pittsburg flux is made by heating petroleum residuum with sulphur. The sample tested was tough and sticky, would cut easily, but would not pull into threads. It melted to a thin liquid at about 195° C. In testing it was mixed with Illinois No. 11 C (4) coal and heated far above 100° C. It produced a satisfactory briquet when 8 per cent was used.

#### ADDITIONAL EXPERIMENTS WITH MIXTURES.

All the briquets made with inorganic binders were brittle, though very hard. Experiment had shown that when brittle pitches, etc., were used, the briquets became less brittle if a thinner pitch or oil was added. Therefore an attempt was made to improve these briquets by the addition of organic binders. For this purpose coal-tar creosote (27), asphalt tar (42), and water-gas tar pitch (39) were chosen. The results are shown in the following table:



*Results of briquetting California and Illinois coals with varying mixtures of organic and inorganic binders.*

Coal.	Binder.				Grade of coher- ence. <sup>a</sup>
	Inorganic constituent.		Organic constituent.		
	Material.	Per cent.	Material.	Per cent.	
Illinois No. 6 B.....	Plaster of Paris.....	6	Coal-tar creosote.....	4	3
	.....do.....	6	Asphalt tar.....	4	4
	.....do.....	6	Water-gas tar pitch.....	4	4
	Portland cement.....	6	Coal-tar creosote.....	4	3
	.....do.....	6	Asphalt tar.....	4	3
	.....do.....	6	Water-gas tar pitch.....	4	4
	Magnesium oxide.....	2	Coal-tar creosote.....	4	3
	.....do.....	2	Asphalt tar.....	4	3
	.....do.....	2	Water-gas tar pitch.....	4	3½
	Plaster of Paris.....	6	.....do.....	8	4½
California No. 1.....	Portland cement.....	6	.....do.....	8	4½
	Magnesium oxide.....	3	.....do.....	8	4
	.....do.....	4	.....do.....	4	3

<sup>a</sup> See p. 22.

The briquets made with Illinois coal and water-gas tar pitch were fairly good and stood up very satisfactorily in the fire. The advantage gained, however, over the use of the water-gas tar pitch alone would not be sufficient to offset the introduction of the 6 per cent of ash with the cement or the plaster of Paris, or the cost of the magnesium oxide when that material is used. The cohesive force of the briquets made with the two binders was no greater than the sum of the cohesive force obtained with each separately. The only advantage to be gained by using such mixed binders would be an added strength in the fire. Experiments with the California lignite were therefore made as above shown. The briquets were found to be considerably improved as to their behavior in the fire by the addition of the inorganic constituent of the binder. Briquets from this coal made with pitch alone fall to pieces badly in the fire. The improvement in this regard, however, is offset by the added expense and the introduction of ash, and it is therefore considered more desirable where possible to mix such noncaking coals with caking coals before briquetting. If this is not practicable then the addition of inorganic binders might be tried as a last resort. Should the inorganic binders be used, magnesium oxide and plaster of Paris will be found to give the most satisfactory results, 3 per cent of the former being equivalent to 5 to 6 per cent of the latter.

### EXPERIMENTS IN BRIQUETTING WITHOUT BINDERS.

Many experiments were made in the endeavor to obtain briquets by heating the coal without binder and then pressing. It was found that if this heating was done in a clay crucible as usual, coherent briquets could not be obtained. But if the heating was done in a small nickel crucible and the pressure applied before the coal was allowed to cool, briquets having considerable coherence were often produced. If the coal cooled after it had softened or commenced to cake, a coherent briquet could not be obtained, and even on again heating the coal it would not cohere in the press. This fact has also been noted by C. C. Catlett.<sup>a</sup> It was undoubtedly because of the necessary chilling of the heated coal in taking it out of the crucible that better results were not obtained by this method. The experiments show the necessity of heating the coal under pressure if briquets are to be made without a binder. The German presses for briquetting lignite coal without a binder, which heat the coal by friction produced in the molds, are undoubtedly based on the right principle.

### RESULTS OF TESTS IN BRIQUETTING DIFFERENT COALS.

The results of the tests here reported should be interpreted in connection with the detailed discussion of each binder. Thus while binders 12 D, 13, 25, 26, etc., mentioned in the table which follows, are too liquid for use as a binder, the pitches or tarry residues left after distilling off the low-boiling oils from these binders will make excellent briquets, as has been already pointed out. It should be remembered, moreover, that the degree of fineness to which the coal is powdered, and also the temperature to which the mixture of coal and binder is heated, will affect the character of the briquet and the percentage of binder necessary to make it coherent. Doubtless an uncontrolled variation in these factors has caused individual results to vary, but probably not to such an extent as to affect any important conclusions to be drawn from the work.

Although many of the binders were tried with only one coal, the result permits the approximate prediction of the percentage of binder for any other coal in the table. It is not possible, however, to predict with the same certainty for the lignites, which show at times variations not susceptible of easy explanation.

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<sup>a</sup> Eng. and Min. Jour., vol. 71, 1901, p. 329.

*Results of tests in briquetting different coals, showing percentages of binder necessary to make a satisfactory briquet.*

Designation of binder.		Field designation of coals and lignites briquetted.											
Material. <sup>a</sup>	No.	Ar- kan- sas. 7 A ?.	Cali- for- nia. 1.	Colo- rado. 1.	Illinois.								
					4.	5.	6 B.	7 C.	7 D.	8.	9 A.	10.	11 B.
1	2	3	4	5	6	7	8	9	10	11	12	13	14
Clay.....	1	8	8	.....	.....	.....	8	.....	.....	.....	.....	.....	8
Magnesia.....	3	4	.....	.....	.....	.....	5	.....	.....	.....	.....	.....	4
Magnesia cement <sup>b</sup> .....	4	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	4
Plaster of paris.....	5	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	12
Portland cement.....	6	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	12
Natural cement.....	7	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	14
Water glass.....	9	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	14
Rosin.....	10	6	6	.....	.....	.....	6	.....	.....	.....	.....	.....	6
Pitch.....	11	3	5	.....	.....	.....	4	.....	.....	.....	.....	.....	3.5
Wood-tar pitch.....	12 A	8	12	.....	.....	.....	12	.....	.....	.....	.....	.....	10
Pine-wood tars.....	12 B	3	8	.....	.....	.....	.....	3	.....	5	5	4	3.5
	12 C	4	9	.....	.....	.....	.....	4	.....	5	5	5	4.5
	12 E	.....	.....	.....	.....	.....	6	.....	.....	.....	.....	.....	.....
Fir tar.....	14	.....	.....	.....	.....	.....	6	.....	.....	.....	.....	.....	.....
Sulphite liquor.....	16	.....	12	.....	.....	.....	10	.....	.....	.....	.....	.....	.....
Cornstarch.....	21	0.5	1	1	.....	.....	1	.....	.....	.....	.....	.....	0.75
Potato starch.....	22	.5	1	1	.....	.....	1	.....	.....	.....	.....	.....	.75
Producer-gas tar pitch.....	24 C	.....	.....	.....	4	.....	.....	.....	.....	.....	.....	.....	.....
Producer-gas tar.....	24 D	4	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
	28 A	6	.....	.....	.....	.....	7	.....	.....	.....	.....	.....	.....
	28 B	8	16	.....	12	8	.....	.....	.....	.....	.....	.....	7
	28 C	6.5	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
	28 D	6.5	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
	28 E	.....	.....	.....	8	.....	.....	.....	.....	.....	.....	.....	.....
	28 F	.....	.....	.....	7	.....	.....	.....	.....	.....	.....	.....	.....
	28 G	.....	.....	.....	8	.....	.....	.....	.....	.....	.....	.....	.....
Coal-tar pitches.....	28 H	.....	.....	.....	6	.....	.....	.....	.....	.....	.....	.....	.....
	28 I	14	.....	20	.....	.....	16	14	.....	16	16	16	15
	28 J	.....	.....	16	.....	.....	12	10	.....	12	12	11	11
	32	.....	.....	.....	3	.....	.....	.....	.....	.....	.....	.....	.....
Maltha.....	37 A	3	6	.....	.....	.....	4	.....	.....	.....	.....	.....	4
	37 C	3.5	.....	.....	.....	.....	4	.....	.....	.....	.....	.....	4
	37 D	.....	.....	.....	6	.....	.....	.....	.....	.....	.....	.....	.....
	37 E	.....	.....	.....	7	.....	.....	.....	.....	.....	.....	.....	.....
Petroleum residuums.....	37 F	.....	.....	.....	6	.....	.....	.....	.....	.....	.....	.....	.....
	39	4.5	8	.....	6.5	.....	6	5	6	6.5	6.5	6	5
Water-gas tar pitch.....	40	3	.....	.....	.....	.....	4	.....	.....	.....	.....	.....	3
Wax tailings.....	41	12	14	.....	.....	.....	14	.....	.....	.....	.....	.....	14
Acid sludge.....	41	12	14	.....	.....	.....	14	.....	.....	.....	.....	.....	14

<sup>a</sup> The following materials were found to be of no use as binders: Lime (2), slag cement (8), wood pulp (15), beet pulp (17), lime cake (18), and impsonite (30). Those found to be too liquid for use as binder were: Pine-wood tar (12 D), pine-wood creosote (12 F), hard-wood tar (13), illuminating-gas tar (25), by-product coke-oven tar (26), coal-tar creosote (27), crude oil (36), water-gas tar (38), asphalt tar (42), and Pintsch gas tar (43). Satisfactory briquets were not obtained from beet-sugar molasses (19) and cane-sugar molasses (20). Blast-furnace tar is not produced in the United States.

<sup>b</sup> Contained sufficient magnesia to make 4 per cent MgO





## GOVERNMENT PUBLICATIONS ON BRIQUETTING.

The following reports (except those to which a price is affixed) can be obtained by application to the Director of the Bureau of Mines, Washington, D. C. The priced publications can be obtained by sending the price, in cash, to the Superintendent of Documents, Government Printing Office, Washington, D. C.

### PUBLICATIONS OF THE UNITED STATES GEOLOGICAL SURVEY.

- BULLETIN** No. 261. Preliminary report on the operations of the coal-testing plant of the United States Geological Survey at the Louisiana Purchase Exposition, St. Louis, Mo., 1904. E. W. Parker, J. A. Holmes, M. R. Campbell, committee in charge. 1905. 172 pp. 10 cents.
- PROFESSIONAL PAPER** No. 48. Report on the operations of the coal-testing plant of the United States Geological Survey at the Louisiana Purchase Exposition, St. Louis, Mo., 1904. E. W. Parker, J. A. Holmes, M. R. Campbell, committee in charge. 1906. In three parts. 1492 pp., 13 pls. \$1.50.
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